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Final Technology Evaluation Report
Volume I

Physical Separation and Acid Leaching:
A Demonstration of Small-Arms Range Remediation
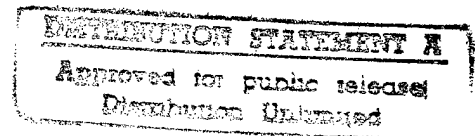# *at Fort Polk, Louisiana*

Prepared for

Naval Facilities   *and*   **U.S. Army**
Engineering   **Environmental**
Service Center   **Center**

by

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Columbus, Ohio

September 22, 1997

19980408 046

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ESTCP

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE September 22, 1997	3. REPORT TYPE AND DATES COVERED Technology Demonstration, Nov 1995- Sep 1997

4. TITLE AND SUBTITLE
Final Technology Evaluation Report Vol. 1
Physical Separation and Acid Leaching: A Demonstration of Small-Arms Range
Remediation at Fort Polk, Louisiana

5. FUNDING NUMBERS

6. AUTHOR(S)
Battelle

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
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505 King Avenue
Columbus, Ohio 43201-2693

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

U.S. Army Environmental Center
Aberdeen Proving Ground,
MD 21010-5401

Naval Facilities Engineering Service Center
1100 23RD Avenue
Port Hueneme, CA 93043-4370

10. SPONSORING / MONITORING AGENCY REPORT NUMBER
SFIM-AEC-ET-CR-97049

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION / AVAILABILITY STATEMENT
Approved for Public Release; distribution is unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*
The U.S. Army Environmental Center in partnership with the Naval Facilities Engineering Services Center and the U.S. Army Engineer Waterways Experiment Station demonstrated Physical Separation/Leaching methods for the remediation of small arms range soils. The demonstration occurred at Fort Polk, Louisiana. After conducting a world-wide search, two vendors were selected to demonstrate two variations of the physical separation/leaching technologies. The first using a process based on acetic (weak) acid chemistry and the second based on hydrochloric (strong) acid chemistry. Following completion of the bench treatability studies, each vendor performed a full scale (5-10 tons per hour, 1000 tons total) demonstration of their respective technologies.

Volume 1 of the Technology Evaluation Report provides a comprehensive description and evaluation of the physical separation/acid leaching technology demonstrated at Fort Polk. Topics covered include: 1) the equipment and processes necessary to accomplish physical separation/acid leaching, 2) the demonstration site and berm soil characteristics, 3) the performance objectives and approach, 4) each vendor's process plant used in the demonstration, 5) the methodology used to evaluate the technologies during the demonstration, and 6) the demonstration results to include: effectiveness, implementability, and cost.
Volume 2 contains the appendices to Volume 1.

14. SUBJECT TERMS
Soil Washing , Heavy Metals, Recycling, Physical Separation, Small Arms Ranges, Technology Demonstration, Acid Leaching, Lead Remediation, Implementation, Maintenance, TCLP, Bench-Scale test, Screening, Attrition Scrubbing

15. NUMBER OF PAGES
144

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT UL

Final Technology Evaluation Report
Volume 1: Main Report

Physical Separation and Acid Leaching:
A Demonstration of Small-Arms Range Remediation
at Fort Polk, Louisiana

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Acknowledgments

Several individuals and organizations participated in this demonstration and provided review, guidance, and information that were valuable to Battelle and NFESC during the evaluation of the physical separation/acid leaching technology at Fort Polk:

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Acronyms and Abbreviations

AA	atomic absorption
AR	Army Regulation
ARAR	applicable or relevant and appropriate requirement
BMRC	Bureau of Mines Research Center
BRAC	Base Realignment and Closure
BZO	battle sight zero
CAAA	Clean Air Act Amendment
CATEX	Categorical Exclusion
cc	concentration criterion
CEC	cation exchange capacity
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
C.I.	confidence interval
CWA	Clean Water Act
DE	diatomaceous earth
DoD	Department of Defense
EDTA	ethylenediaminetetraacetic acid
EPA	United States Environmental Protection Agency
EPCRA	Emergency Planning and Community Right-to-Know Act
EQCC	Environmental Quality Control Committe
ESTCP	Environmental Security Technology Certification Program
FUDS	Formerly Used Defense Site
HASP	Health and Safety Plan
HSO	Health and Safety Officer
ICP-AES	inductively coupled plasma atomic emission spectroscopy
ISV	in situ vitrification
NAAAQS	National Ambient Air Quality Standards
NCEL	Naval Civil Engineering Laboratory
NEPA	National Environmental Policy Act
NESHAPS	National Emission Standards for Hazardous Air Pollutants
NFESC	Naval Facilities Engineering Service Center
NPDES	National Pollutant Discharge Elimination System
NRA	National Rifle Association
NSPS	New Source Performance Standards

O&M	operations and maintenance
OPS	operations
OSHA	Occupational Safety and Health Act
PAH	polycyclic aromatic hydrocarbon
POTW	publicly owned treatment works
PPE	personal protective equipment
PRP	potentially responsible party
PSD	Prevention of Significant Deterioration
QA	quality assurance
QC	quality control
RAB	Remediation Advisory Board
RCRA	Resource Conservation and Recovery Act
REC	Record of Environmental Consideration
RPM	Remediation Program Manager
RSD	relative standard deviation
SARA	Superfund Amendments and Reauthorization Act
SEM	scanning electron microscope
SITE	Superfund Innovative Technology Evaluation
SPCC	Spill Prevention, Control, and Countermeasures
S/S	solidification/stabilization
STLC	Soluble Threshold Limit Concentration
SVOC	semivolatile organic compound
SWPP	Solid Waste Pollution Prevention
TCLP	Toxicity Characteristic Leaching Procedure
TOC	total organic carbon
TPQ	threshold planning quantity
TSD	Treatment, Storage, and Disposal
TSDF	treatment, storage, and disposal facility
TTLC	Total Threshold Limit Concentration
USAEC	U.S. Army Environmental Center
VOC	volatile organic compound
WET	Waste Extraction Test (of California)
XRD	x-ray diffraction
XRF	x-ray fluorescence

Physical Separation and Acid Leaching:
A Demonstration of Small-Arms Range Remediation at Fort Polk, Louisiana

by

Battelle Columbus Operations

September 22, 1997

1. Introduction

1.1 PROJECT BACKGROUND

Approximately 2,600 United States Department of Defense (DoD) installations have small-arms training ranges which contain soil contaminated with heavy metals, particularly lead. These metals may accumulate in the range soils to the extent that the site encounters ricochet problems or a potential for migration of heavy metals to surface waters or groundwater. Maintenance or remediation of these sites currently involves contaminant stabilization and/or landfilling. These methods minimize the hazards due to contaminant migration and exposure to humans and the environment, but the contamination and the liability for it remain. In addition, processes such as stabilization can significantly increase the volume of the final waste form.

New processes are needed that are cost competitive with the stabilization/landfilling processes, result in removal of the heavy metal contaminants, and provide substantial net waste volume reduction. Moreover, processes that remove lead from the soil are also required in order to support the reuse of the site for beneficial purposes. Physical separation and acid leaching are

processes developed partly by the mining industry for recovery of metal values from ores. The application of these processes for the removal of heavy metals from small-arms range soils has recently been explored in bench- and pilot-scale studies. The attractiveness of this approach stems from the fact that metals of concern are removed from the range soils and may even be recycled off site. The site can be restored to a wider range of beneficial uses.

1.2 OFFICIAL DOD REQUIREMENT STATEMENT(S)

The Environmental Security Technology Certification Program (ESTCP) sponsored the U.S. Army Environmental Center (USAEC) and the Naval Facilities Engineering Service Center (NFESC) to conduct a joint demonstration of small-arms range remediation at Fort Polk. Figure 1-1 shows the various participants and their roles. The main requirement of this demonstration was to evaluate and document the performance and cost of two physical separation/acid leaching processes that can be used to process small-arms range soils at DoD sites. Battelle was contracted by the Naval Facilities Engineering Service Center (NFESC) to conduct an independent evaluation of the two processes and their implementation at Fort Polk. The methodology for the evaluation is documented in the Environmental Security Technology Certification Program (ESTCP)-approved Technology Demonstration Plan prepared by Battelle (1996).

1.3 OBJECTIVES OF THE DEMONSTRATION

The primary goal for the demonstration was to evaluate the technical and economic performance of physical separation and acid leaching for the removal of lead from small-arms range soils. Two vendors demonstrated their variations of physical separation and acid leaching on Range 5 berm soils at Fort Polk to remove the heavy metal contamination. Vendor 1's leaching process was based on acetic (weak) acid chemistry. Vendor 2's process was based on hydrochloric (strong) acid chemistry for the leaching process. The goal was not to compare the two vendors, but to evaluate the suitability of the two acids for processing of small-arms range soils.

The following performance objectives were specified for each vendor:

❏ Process 1,000 tons of Range 5 berm soils during the demonstration. Complete the mobilization, operation, and demobilization phases of the demonstration within a 3-month period. (Each vendor was expected to keep the plant operational for a period of 15 days.)

❏ Demonstrate the technology at a continuous processing rate of 5 to 10 tons per hour.

❏ Meet the total lead criterion of 1,000 mg/kg or less in the processed soil. This criterion was changed to 500 mg/kg for Vendor 2 to better meet the Toxicity Characteristic Leaching Procedure (TCLP) criterion.

❏ Make a good faith attempt to meet the TCLP lead soil criterion of 5 mg/L for the processed soil.
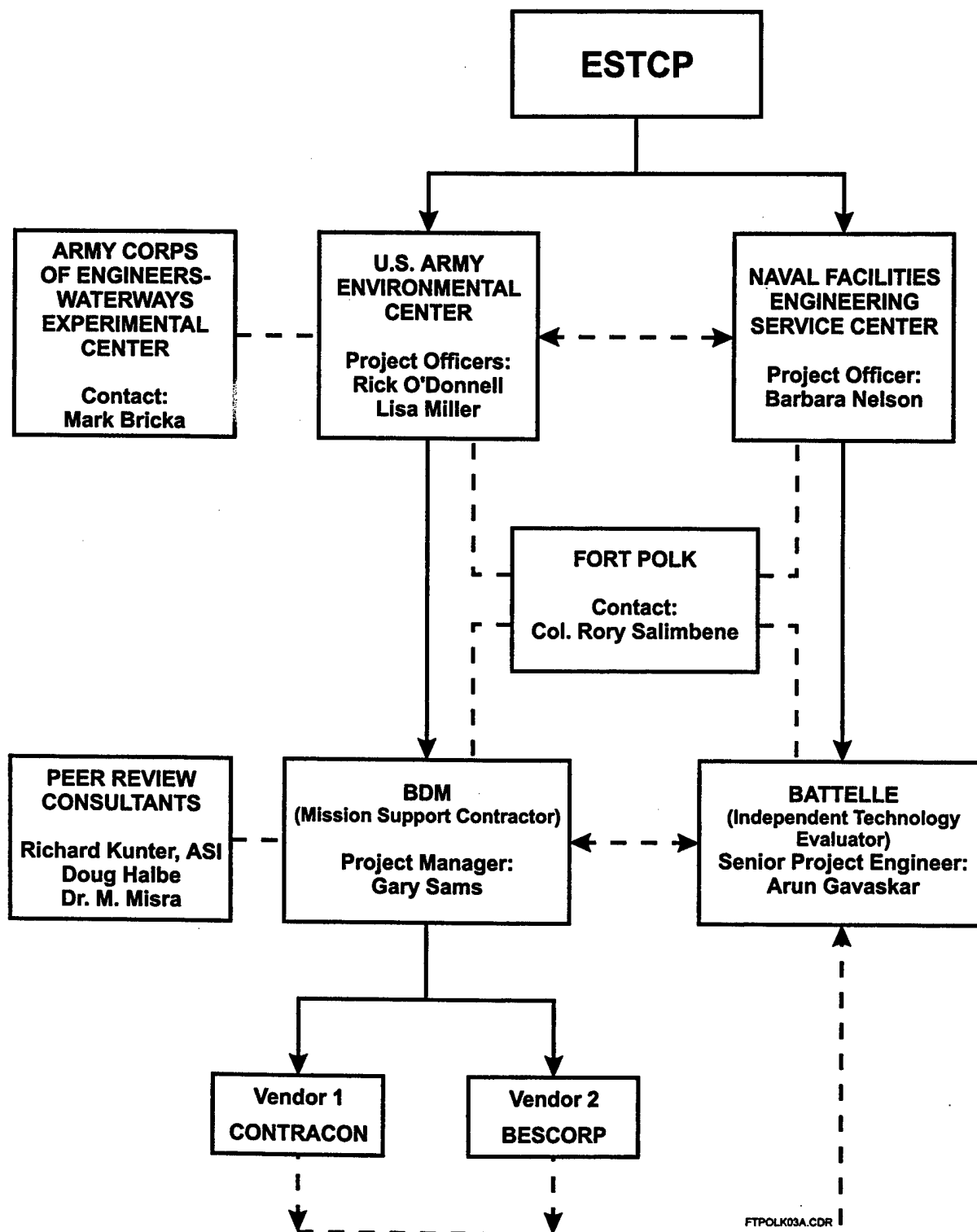
Figure 1-1. Overall Project Organization

☐ Ensure that the processed soil is otherwise acceptable (physically and chemically) for returning Range 5 to its former useful condition.

☐ Minimize residuals requiring off-site disposal. Ensure acceptability for off-site recycling of lead concentrates.

Battelle evaluated the performance of the technology and its implementation at Fort Polk through preparation of a Technology Demonstration Plan (Battelle, 1996), on-site observation and sampling during the field operation, discussions with vendors and site support personnel, off-site laboratory analysis of samples from various process streams, and evaluation of field and laboratory data.

1.4 TECHNOLOGY DESCRIPTION

The combination of physical separation and acid leaching is an innovative remedial alternative that has received increasing interest (van Benschoten et al., 1997). Physical separation is a technique for dividing soil into different size or density fractions. Physical separation rarely produces material that is sufficiently clean to allow reuse or disposal directly, but works well as a pretreatment so that the volume of soil requiring leaching is reduced. When particulate contaminants are present, physical separation reduces the contaminant load on the leaching process. Section 2 provides a detailed description of various physical separation and leaching techniques.

Physical separation and acid leaching are particularly useful at sites where metallic contaminants are present as particulates, e.g., small-arms ranges or battery recycling sites. First, oversize debris, such as rocks, that typically have low concentrations of metals is removed. This debris fraction can usually be cleaned easily by washing or leaching with a dilute acid solution. Metal fragments are then separated from the bulk soil based on particle size and density. The separated metals stream may be suitable for off-site recycling. The lighter smaller soil that remains consists of sands, silts, and clay and may also contain very fine metal particulates and bound molecular or ionic metals. The soil particles and associated heavy metal contaminants can be effectively treated with acid leaching. Different extractants may be used depending on the physical and chemical form of the heavy metals and the matrix characteristics.

1.5 REGULATORY ISSUES

The following regulations need to be addressed for the application of physical separation/acid leaching technologies to small-arms range maintenance or remediation activities:

☐ National Environmental Policy Act (NEPA)

☐ Resource Conservation and Recovery Act (RCRA)

☐ U.S. Environmental Protection Agency's (EPA's) Military Munitions Rule and the DoD's Military Range Rule

- ☐ Emergency Planning and Community Right-to-Know Act (EPCRA)

- ☐ Clean Water Act (CWA)

- ☐ Clean Air Act Amendment (CAAA)

- ☐ Occupational Safety and Health Act (OSHA)

- ☐ State and local regulations (e.g., Title 22, California Code of Regulations).

These regulations and their application are discussed in Section 9. In general, removal of heavy metals is expected to be conducted as a maintenance activity at active ranges and as a remediation activity at inactive ranges.

1.6 PREVIOUS TESTING OF THE TECHNOLOGY

Appendix E-2 describes previous bench-scale, pilot-scale, and commercial applications of physical separation and acid leaching.

2. Physical Separation and Acid Leaching Principles

This section describes the types of equipment and processes used to accomplish physical separation and acid leaching for treatment of metal contaminants in soils.

2.1 PHYSICAL SEPARATION PROCESSES DESCRIPTIONS

This subsection provides background information of a variety of commonly used physical separation methods. Physical separation techniques have been used commonly in the chemical and mining industries for many years. These techniques involve the physical separation of particles from each other based on particle characteristics such as size, shape, density, or magnetism. Five classes of physical characteristics provide a practical basis for separating particles:

- ❏ Particle size (screening)
- ❏ Particle hydrodynamics (settling velocity)
- ❏ Particle density (gravity separation)
- ❏ Surface properties of particles (flotation)
- ❏ Magnetic properties (magnetic separation).

The attributes of these common particle separation techniques are summarized in Table 2-1.

Table 2-1. Key Attributes of Common Particle Separation Techniques

	Technique				
	Size Separation (Screening)	Hydrodynamic Separation (Classification)	Density (Gravity) Separation	Froth Flotation	Magnetic Separation
Basic Principle	Various diameter openings allow passage of particles with different effective size	Different settling rates due to particle density, size, or shape	Separation due to density differences	Particles are attracted to bubbles due to their surface properties	Magnetic susceptibility
Major Advantage	High-throughput continuous processing with simple, inexpensive equipment	High-throughput continuous processing with simple, inexpensive equipment	High-throughput continuous processing with simple, inexpensive equipment	Very effective for fine particles	Can recover a wide variety of materials when high gradient fields are used
Limitations	Screens can plug; fine screens are fragile; dry screening produces dust	Difficult when high proportions of clay, silt, and humic materials are present	Difficult when high proportions of clay, silt, and humic materials are present	Particulate must be present at low concentration	High capital and operating cost
Typical Implementation	Screens, sieves, or trommels (wet or dry)	Clarifier, elutriator, hydrocyclone	Shaking table, spiral concentrator, jig	Air flotation columns or cells	Electromagnets, magnetic filters

Sources: U.S. EPA, 1995, EPA/540/R-95/512.

2.1.1 Size Separation

Segregating solids according to particle size, called screening or sieving, is done by passing the solids through a screen formed by a wire mesh with specifically sized openings. The oversize fraction tends to remain on the screen and the undersize fraction tends to pass through, but the separation is not absolute. Larger particles may pass through to the undersize fraction, if the particle shape is highly asymmetric. Smaller particles may be retained in the oversize fraction due to blocking of some screen opening or physical attachment to larger particles. The oversize fraction, if allowed to build up on the screen, can block the openings. Therefore, screens often are sloped to allow the oversize fraction to roll off. Screens can either be stationary or have some motion (shaking, vibrating, or gyrating) to dislodge particles that block the openings.

Screening can be conducted either dry or wet. In dry screening, the soil is placed directly onto the screen without any water addition. In wet screening, the soil is wetted by mixing with water to form a slurry before screening or by spraying water on the screen to dislodge blocking particles.

2.1.1.1 Dry Screening

Screening of dry soil invariably is required at most soil remediation sites, if nothing else, to remove rocks, branches, or other oversize material from the bulk of the soil. Dry screening is effective on large to intermediate size ranges as long as the feed material is essentially dry. However, this condition is rarely satisfied in field operation, where natural moisture makes dry screening difficult below ½ inch or ¼ inch because of clogging. If finer screening is required, the soil must be dried before screening; alternatively, wet screening can be applied.

2.1.1.2 Wet Screening

In soil remediation, caution should be exercised before selecting a wet separation technique, which invariably generates a water stream that must be treated before discharge. Although a dewatering step allows the water to be recirculated, the user is still left with a wastewater stream after the last batch of soil has been treated. The other consideration is that wet separation processes may leave the user with wet soil (25 to 75% solids) that is difficult to handle in down-stream chemical treatment. Clay soils especially become very difficult to handle and may stick to equipment when wet. Therefore, wet separation should be conducted only after weighing its potential benefits. The following guidelines should be followed in deciding whether or not to use wet separation:

❑ Wet separation is most worthwhile if a sizable fraction of the metal contamination is particulate. In that case, wet separation may either render the soil nonhazardous (not requiring further treatment) or reduce the quantity of contaminant particles to a level where significantly smaller amounts of treatment chemicals are required downstream.

❑ Wet separation is worthwhile if the chemical treatment that follows benefits from water addition, e.g., soil washing or heap leaching.

In addition, wet classification could still be beneficial if the metal fraction recovered is recyclable or if downstream chemical treatment requirements are significantly reduced.

2.1.2 Hydrodynamic Separation (Classification)

Hydrodynamic separation, or classification, is a technique of separating particles into two or more fractions based on the velocity of particles moving through a viscous fluid. A particle's velocity in the fluid is determined by the particle's size, density, and shape. Separation often is enhanced by keeping the fluid in motion in a direction (upward) opposite to that of the falling particles.

When the particle size falls below that required for efficient screening (usually about 200 μm), classification is used. As with screening, separation by classification depends on particle size; however unlike screening, classification also depends on particle density. Wet classifiers (hydro-classifiers) are more common than air classifiers. Classifiers operate over a wide range of particle sizes. Large elutriators have been used in the past to separate lead particles (from car batteries) several millimeters in diameter from junkyard waste. Other classifiers, such as spiral classifiers and settling cones, are used for desliming, i.e., removing very fine particulates from a slurry. The hydrocyclone classifier efficiently separates very fine particles and has been used to deslime, degrit, and dewater (thicken). Hydrocyclones most commonly are used on particles in the 150- to 5-μm size range, although coarser materials can be separated. Hydrocyclones are relatively small, inexpensive devices. A cyclone bank (group of cyclones in parallel) is used for higher capacities.

2.1.2.1 Elutriator

An elutriator consists of a vertical column containing water flowing from the bottom to the top. Soil to be processed is introduced at the top or part way down the column. The falling particles reach their terminal velocity based on their size, shape, and density. The water flow into the bottom of the column is adjusted such that particles having a terminal velocity less than the water velocity are carried up by the rising water stream. The mixture of water and finer, lighter particles is called slimes or tailings. Larger, heavier particles settle fast enough to overcome the water velocity and travel down the column. Desired fractions of the settling particles (middlings and concentrate) can be collected at different depths along the column. Alternatively, a series of sorting columns, each with a different water velocity, can be used to obtain the desired fractions.

2.1.2.2 Mechanical Classifier

Hydrodynamic separation can also be accomplished by mechanical action in a mechanical classifier were a soil water slurry is introduced into an inclined trough. Coarse particles quickly settle out of the slurry and fall to the bottom. The slimes overflow from the lower end of the trough. The coarse particles are carried up the incline by a rake (rake classifier) or spiral (spiral classifier or "sand screw") and discharged, as shown in Figure 2-1.
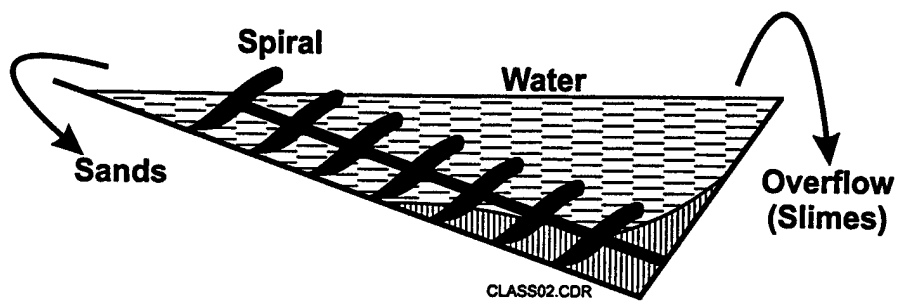
Figure 2-1. Schematic of a Spiral Classifier or "Sand Screw"

2.1.2.3 Hydrocyclone

A hydrocyclone is a continuously operating device that uses centrifugal force to accelerate the settling rate of particles (Figure 2-2). The hydrocyclone consists of a vertical cone into which the feed (in the form of a slurry) is introduced tangentially at the top. A vortex is created with a low-pressure zone along the vertical axis of the cone. Faster settling particles (those having large size or higher density) are accelerated to the wall of the cyclone by centrifugal force, and move in spiral form along the wall down to the bottom opening. The slower-settling particles (fines) are
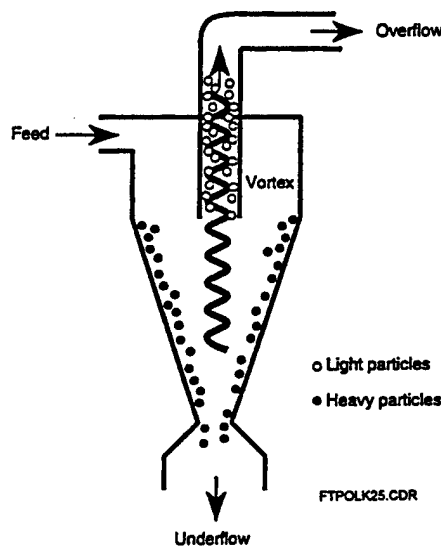
Figure 2-2. Hydrocyclone

drawn to the low-pressure zone along the axis and pulled out at the top through a central tube called the vortex finder. Hydrocyclones often are very small devices. For higher throughput, a group of hydrocyclones (hydrocyclone bank) are configured in parallel.

2.1.2.4 Attrition Scrubbing

Attrition scrubbers are often used to pretreat the raw soil feed before size or density separation. Many attrition scrubbers are structured somewhat like the classifier shown in Figure 2-1. These scrubbers break up soil agglomerates into individual particles and "scrub" oxide or other coatings from individual particles. Soil scrubbing is accomplished mostly by particle-to-particle abrasion or attrition, but also by the interaction between equipment parts (e.g., paddles or propellers) and the particles. Some attrition scrubbers intensively mix and scrub materials with two large-diameter propellers that are oppositely pitched and enclosed in a cell. Baffles are sometimes included in the cells to direct the flow of material. Single- or multiple-cell designs are available for the required throughput.

A log washer consists of an inclined trough that surrounds two shafts fitted with paddles. The incline reduces the transport effect of the paddles and increases the mass weight against the paddles. The paddles are pitched to convey materials to the discharge end against the incline of the trough. The name "log washer" is a misnomer because it implies that the machine is designed to wash logs. The name actually comes from the fact that the first units used to wash sand and gravel incorporated wooden logs as the shafts, with steel paddles set into them. In the aggregate business, the log washer is known best for its ability to remove tough, plastic clays from natural and crushed gravel, crushed stone, and ore feeds.

A blade mill is similar to a log washer in design, and is used to perform the same function. Unlike a log washer, a blade mill has just one shaft. Because of the single-shaft design, a blade mill may impart less energy to the material being washed than a log washer. Log washers and blade mills are designed to handle larger particle sizes than can be accommodated by conventional attrition scrubbers.

2.1.3 Density (or Gravity) Separation

Gravity concentration methods separate particles mainly on the basis of their density. Particles with different densities respond differently to gravity and to one or more other forces applied simultaneously in opposition to gravity. Although density difference is the main criterion for gravity separation, particle size and shape also influence the separation. In general, gravity separation is more efficient with coarser particles.

Techniques using gravity concentration are efficient down to the 50- or 10-μm range, and high throughputs are possible using relatively small equipment. Among gravity concentrators, jigs can separate out relatively coarse material ranging in size from ½ inch down to 150 μm. Relatively good recoveries are possible even down to 75 μm. Performance is better with either (1) a high-particle-density difference in an unclassified feed (wide size range) or (2) a low-particle-density difference in a classified feed (narrow size range).

2.1.3.1 Jig

The jig, one of the oldest gravity separation devices, achieves particle separation using a pulsating water column as shown in Figure 2-3. A mixture of soil and water (10 to 60% solids) is subjected to vertical flow pulsations that alternately lift and lower the entire mass (water and particles). The upward -pulse tends to loosen the bed of particles, whereas the downward pulse tends to consolidate the bed. The heavier particles make their way progressively to the bottom with each pulse cycle. The downward movement of the lighter particles is retarded by the upward pulse but is not accelerated fast enough by the downward pulse. A bed of heavier particles soon builds at the bottom and the lighter particles go with the overflow. A bed of ragging (usually made of steel balls) further retards the downward movement of lighter particles.
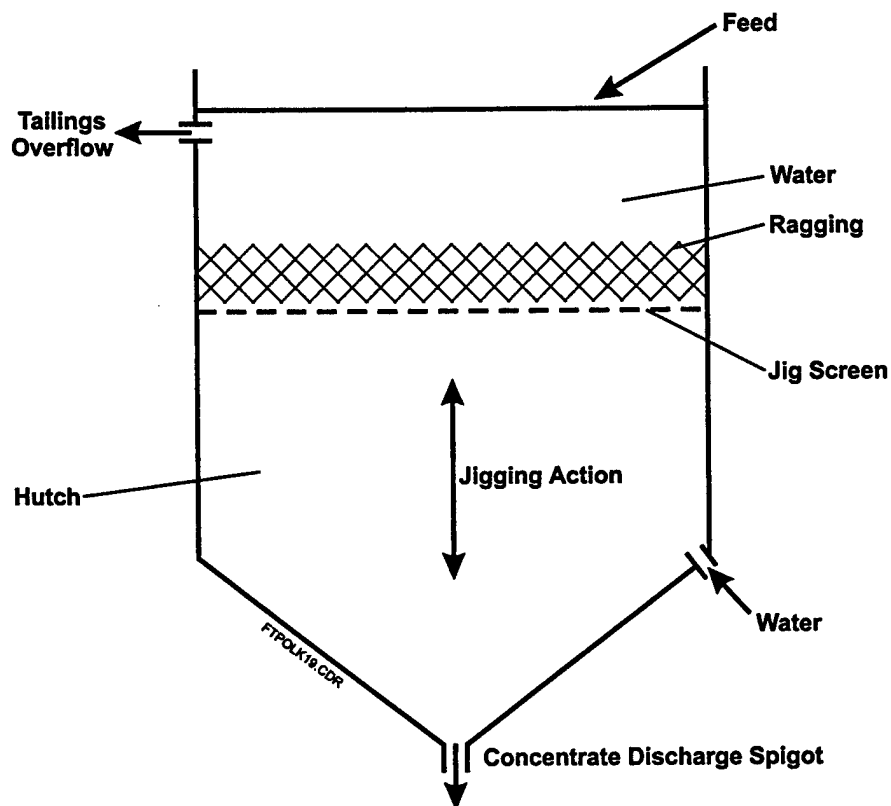
Figure 2-3. Basic Jig Construction

2.1.3.2 Spiral Concentrator

The spiral concentrator is another popular type of gravity separator. This device consists of a helical channel that winds down a central pole. Feed is introduced at the top of the spiral as a 10% to 40% solids slurry. As the slurry flows down the spiral, a velocity gradient is created along the thickness of the water film. The water closest to the channel surface flows very slowly due to friction, whereas the velocity increases toward the top of the water film as shown in

Figure 2-4. The smallest particles submerge in the slower moving layer of the film. The larger particles and the bulk of the fluid are faster moving and are subjected to centrifugal force along the curved path, which causes them to move outward. The smallest, densest particles, in trying to take the shortest path down, move to the axis of the spiral where they form a band. Ports collect the dense material at several points along the downward path of this band. The width of the concentrate band removed at the port is controlled by adjustable wedges (called splitters).
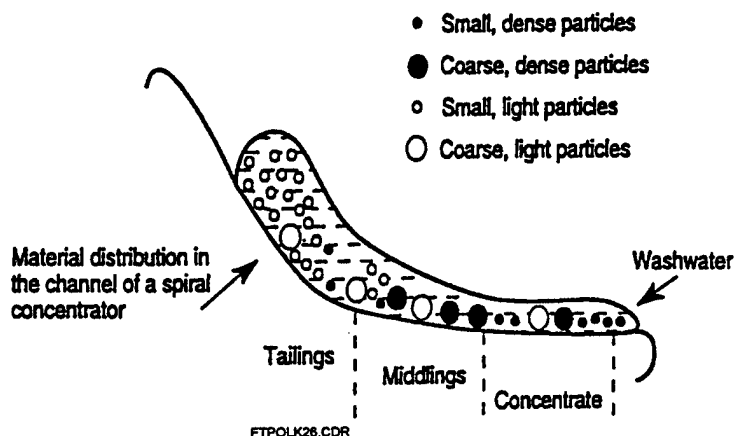
Figure 2-4. Cross Section of a Spiral Channel in a Spiral Concentrator

The most concentrated product goes to the highest port in the spiral, and concentration quality declines as the channel winds downward. The lighter particles are carried outward by the faster moving water at the outer edge of the spiral and descend to the bottom as tailings. A middlings product can be isolated between the concentrate band and the tailings band. Because the concentrate band is very high in solids and the bulk of the water flows to the outer edge of the channel, washwater has to be continuously introduced along the spiral to keep the band moving.

Spiral concentrators are used in the size range of 3 mm to 75 μm. The high capacities can be further increased in the same space by winding two channels down the same column (double spiral).

2.1.3.3 Shaking Table

The shaking table operates according to a principle similar to that of the spiral concentrator. This device consists of a slightly inclined deck to which a 25% solids slurry is introduced at the higher corner. The flowing film separates the small dense particles (which move quickly to the lower, slower-moving layer of the film) from the coarse, light particles as shown in Figure 2-5. The effect is enhanced by vibrating the table at right angles to the water flow in a slow forward stroke and a fast return stroke. The net effect is that the particles move diagonally across the table. Stratification is enhanced by riffles that run along the long axis of the table parallel to the vibrations. The small, dense particles settle down quickly into the riffles near the feed end. These particles travel along the riffles to the side of the table. The coarser, lighter particles go over the

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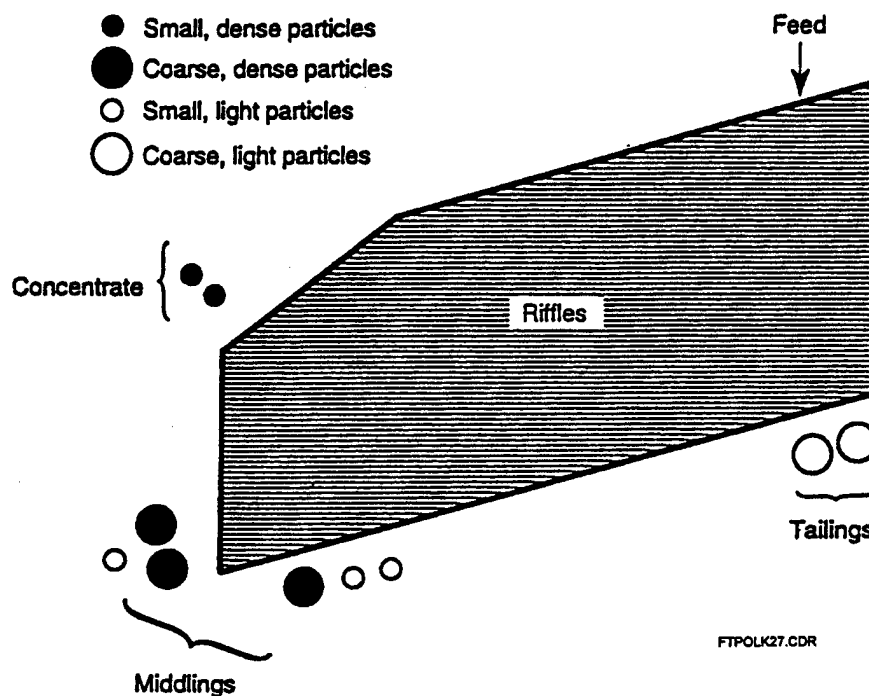
Figure 2-5. Schematic of Shaking Table

riffles to the front of the table. Concentrate, middlings, and tailings can be isolated as required by adjustable splitters placed along the edges of the table.

Shaking tables effectively separate coarse, light particles from fine, dense particles. Therefore, the feed is first classified, because classifiers put these two types of particles in the same product. Sands tables operate on feed sizes between 3 mm to 100 μm. Slimes tables operate on sizes below 100 μm.

2.1.3.4 Bartles-Mozley Table

The Bartles-Mozley Table is used for particles in the size range from 100 μm all the way down to 5 μm. On a normal table, such a small particle size would require a very large surface area. However, this table combines a very high capacity in a very small space with low power consumption.

2.1.4 Froth Flotation

Flotation was developed in the early 1900s as a way of recovering metal value from low-grade ores that otherwise would have been discarded as uneconomical to process. Particle separation by froth flotation is based on the fact that different minerals have different surface properties. These differences in surface properties can be accentuated by adding suitable chemicals to a

slurry containing the minerals. Air is sparged from the bottom of a tank or column containing the slurry. One type of mineral selectively attaches to the air bubbles and rises to the top and forms froth that can be collected. Successful flotation depends on the mineral surface being somewhat hydrophobic so the air bubbles attach. Also, if the bubbles are to continue to hold the mineral at the top, the froth formed should be stable. These characteristics are imparted by the addition of chemicals called flotation reagents.

2.1.5 Magnetic Separation

Magnetic separation is based on the differences in magnetic properties of the various minerals, especially for separating ferrous from nonferrous materials. Commercial units generally operate continuously by subjecting a moving stream of particulates (transported by a conveyor belt or drum) to a strong magnetic field.

2.1.6 Dewatering

With the exception of dry screening, physical separation techniques use water to facilitate transfer and separation of the solid particles. Dewatering often is required to recover and reuse water. It is important to recover this water because it may contain elevated levels of soluble and suspended metals. Commonly used processes for dewatering include filtration, expression, centrifugation, and sedimentation (or thickening). These four dewatering techniques are summarized briefly in Table 2-2. A combination of these methods typically is used to obtain successively drier solids.

2.1.6.1 Filtration and Expression

The process of filtration involves passing a slurry through a porous medium in which the solids are trapped and the liquid goes through. The process of expression involves compressing the liquid, with liquid escape through a porous filter. In any filtration or expression application, a cake of solids gradually forms on the filter media that resists the flow of water. This resistance is overcome by applying a pressure field upstream of the filter or a vacuum downstream from the filter. Filtration equipment is available in several different types. The most common is the filter press, which consists of a row of plates and frames placed alternately. A filter cloth covers each plate. The slurry is introduced into the empty frames. The plates are then pressed toward each other by a screw or hydraulic piston. The water gets squeezed through the filter cloth into grooves in the plate and is removed. The filter cake in the frames can be washed, and the plates can be separated one by one to discharge the cake. The filter cloth is often precoated with a filter aid, such as diatomaceous earth, to prevent clogging.

2.1.6.2 Sedimentation

The settling of solid particles in water is called sedimentation. Because very fine particles settle very slowly, coagulants or flocculants must be added to agglomerate the particles to improve the settling rate. Sedimentation is performed in specialized tanks called either clarifiers or thickeners, depending on the main performance objective. Clarifiers are used if the objective is to

Table 2-2. Key Attributes of Common Dewatering Techniques

Technique	Filtration	Expression	Centrifugation	Sedimentation
Basic Principle	Passage of particles through porous medium based on particle size	Compression with liquid escape through porous filter	Artificial gravity settling: particle size, shape, density, and fluid density	Gravity settling: particle size, shape, density, and fluid density; aided by flocculants
Advantages	Simple operation, more selective separation	Handles slurry materials that are difficult to pump Produces solid with low water content	Fast, large capacity	Simple, less expensive equipment; large capacity
Limitations	Batch nature of operations, washing may be poor. Clogging could be a problem	High pressures required, high resistance to flow in cases	Expensive, more complicated equipment	Slow
Example Equipment Types	Drum, disk, horizontal (belt) filters	Batch and continuous pressure	Solid bowl sedimentation and centrifugal, perforated basket	Cylindrical continuous clarifiers, rakes, overflow, lamella, deep cone thickeners
Typical Bench Test Equipment	Vacuum filters, filter press	Filter press, pressure equipment	Bench or floor centrifuge	Cylindrical tubes, beaker, flocculants

remove solids from liquids, and the clarified liquid is decanted from the top. Thickeners are used if the objective is to remove liquids from solids. The settled solids can be thickened continuously by introducing the feed slurry to the center of the tank, letting the clear liquid overflow over the sides, and evacuating the thickened sludge from the bottom.

2.1.6.3 Centrifugation

The process of centrifugation involves the use of a centrifugal force created by a rotating bowl instead of gravity to bring about separation. For continuous operation, a bowl centrifuge can be used. The solids settle out on the sides of the bowl and the screw conveyor discharges them at the smaller end. A slightly different type of centrifuge is the basket centrifuge, in which solids move to the sides of the rotating basket and are trapped there, whereas the water flows through.

2.2 APPLICATION OF PHYSICAL SEPARATION PROCESSES TO SMALL-ARMS RANGE SOILS

Physical separation is a simple, low-cost method to separate contaminated soils by size, density, or magnetic properties. These separations do not have a high degree of selectivity. Physical separation can make preliminary differentiation that reduces the volume of material treated or helps to optimize operation of subsequent steps but usually is not sufficient to separate clean soil from contaminants.

Physical separation is ideally suited to treatment of small-arms range soils. Both the density differences and size characteristics ease physical separation of bullet metal contaminants from soils. The lead and copper alloy fragments and oxides have a significantly higher density than soil materials. Many bullets will remain sufficiently intact to be larger than soil materials unless there is a lot of gravel. A basic dry screening step to remove bullets that have remained largely intact (generally 0.25 to 0.5 inch diameter in size) is a first step in treatment of soils from most small-arms range berms or impact areas. The smaller bullet fragments are similar in size to soil particles. Separating these smaller fragments from soils requires more complex physical separation steps, but may still be economical. Bullet cores are typically 99% lead and bullet jackets are typically 90% copper.

The applicability of physical separation techniques depends, to a large extent, on particle size. The size ranges suitable for the various techniques are shown in Table 2-3. As seen in this table, many of these techniques have good applicability in the intermediate size range (between 100 and 1,000 μm). Very few techniques are applicable in the fine particle range. In the case of froth flotation, there is an upper limit on the size range based on the size (or weight) of the particle that the air bubbles are capable of supporting. Because soil usually contains a wide range of particle sizes and the performance of physical separation techniques depends on particle size, there are often situations in which a single technique will not achieve sufficient separation. In that case, a combination of techniques may be able to achieve the desired separation. The particle-size ranges shown in Table 2-3 can be used to determine which separation technique(s) should be used.

Table 2-3. Particle-Size Range for Application of Separation Techniques

Separation Process	Particle-Size Range
Size Separation	
Dry screen	>3,000 μm
Wet screen	>150 μm
Hydrodynamic Separation	
Elutriator	>50 μm
Hydrocyclone	5 to 150 μm
Mechanical classifier	5 to 100 μm
Density Separation	
Jig	>150 μm
Spiral concentrator	75 to 3,000 μm
Shaking table	75 to 3,000 μm
Bartles-Mozley Table	5 to 100 μm
Froth flotation	5 to 500 μm

Adapted from: Perry and Chilton (1984) and Wills (1985).

The performance of physical separation techniques depends on the size range and density difference of the feed material. The raw berm soil should be characterized to find the particle size range of the soil and the contaminant distribution within each size range. Size distribution can be determined readily in a laboratory by passing a small sample of air-dried soil from the site through a series of standard sieves. At some sites that have wet, sticky clay, attrition scrubbing and wet sieving are required to ensure that clay balls do not roll off with the oversize fraction. Each size fraction is then subjected to a chemical (metals) analysis to determine the distribution of the contaminants among various size fractions.

If the contamination is particulate, the density difference between the soil and contaminant particles is determined. If the density difference is significant, classification followed by gravity concentration techniques will perform well. Actual recoveries cannot be predicted without tests on site-specific soils; however, the efficiency of separation can be estimated by the following "concentration criterion" (cc) (Wills, 1985):

$$cc = \frac{S_h - S_f}{S_l - S_f}$$

where S_h = specific gravity of heavy particles (usually metal contamination)

S_f = specific gravity of separation fluid medium (usually water)

S_l = specific gravity of light particles (usually soil)

If cc is greater than 2.5, gravity separation can be expected to perform well. Between 1.25 and 2.5, separation should still be feasible; below this the separation may not be feasible. Calculated concentration criteria for various elements and compounds typically found in small-arms range soils are shown in Table 2-4. As seen from the high cc values in the table, the particulate metal oxides and carbonates considered are amenable to gravity separation.

The efficiency of gravity separation also increases with particle size, because larger particles respond better than smaller particles. For a given value of the concentration criterion, the size

Table 2-4. Illustration of Calculated Concentration Criteria for Gravity Concentration

Heavy Material	Heavy Material Specific Gravity (S_h)[a]	Concentration Criteria for Various Combinations of Specific Gravity Light Material Specific Gravity(S_l)[a]		
		2.2	2.4	2.6
Copper metal, Cu	8.96	6.6	5.7	5.0
Cupric oxide, CuO	6.4	4.5	3.8	3.4
Cuprous oxide, Cu_2O	6.0	4.2	3.6	3.1
Lead metal, Pb	11.3	8.6	7.4	6.4
Cerussite, $PbCO_3$	6.5	4.6	3.9	3.4
Lead oxide, PbO[b]	9.3	6.9	5.9	5.2

(a) Specific gravity values used to illustrate light silica soil particles.
(b) Amorphous form.

fraction containing most of the contamination will control the separation performance. Good size control through the judicious use of screens and classifiers before gravity concentration will enhance the efficiency of the separation. Small particulate size solids reduce the processing rate and/or separation efficiency of gravity separation and should be removed prior to gravity separation.

Other equipment-related variables can be adjusted to improve performance. For example, one of the most important variables is the water balance in the separation scheme. Most gravity concentrators have an optimum solids level for the feed slurry. Good solids level control is important, especially for the initial feed. As the material travels through the separation scheme, water can be added or removed as required with the use of washwater lines or thickeners and hydrocyclones.

In jigs, the density effect can be accentuated compared with the size effect by using a short jigging cycle (i.e., short, fast strokes). The short cycle allows smaller, denser particles to be affected more by initial acceleration (mass effect) rather than by terminal velocity (size effect). For coarser particle sizes, longer, slower strokes are better. Similarly, separation in spiral concentrators can be improved by selecting a spiral with a suitable channel slope. Manufacturers of spiral concentrators produce equipment with different channel slopes. For most applications, the purchaser can select an optimum slope from the standard equipment. Gentler slopes are provided for smaller density differences, but with a concomitant drop in capacity. Steeper slopes are for larger density differences and larger throughputs. The performance of tables is most affected by particle size. The wider the particle size range of the feed, the lower the performance. Table performance can be affected also by adjusting the stroke. A shorter stroke with higher speed improves the separation of finer particles; a longer stroke with slower speed is suitable for coarser particles.

Particle size also is important in froth flotation, because air bubbles will not lift particles when the particle weight is sufficiently high to overload the forces of adhesion at the bubble-particle interface. Another factor affecting flotation performance is pH. A higher pH generally is more suitable to flotation, because most collectors are stable in this range. Alkalinity is maintained by the addition of lime.

At some sites, physical separation alone may be sufficient to meet processing targets, especially for range maintenance. At other sites, chemical processing may be necessary to remove additional heavy metals.

2.3 ACID LEACHING PROCESS DESCRIPTION

After physical separation, most of the coarse particulate metals have been removed from the bulk soil. Lead and other metals are still present in the soil either as fine particulates or as molecular or ionic species bound to the soil matrix. Fine particulates could consist of either elemental lead or precipitates of lead salts. Lead species could be bound to the soil by ion exchange, sorption, or complexation with organic matter. Because sorption and ion exchange are surface phenomena, molecular or ionic lead species often are concentrated in the finer size fraction (silts and clays), which have a high surface area.

Soil washing is a group of techniques that try to mobilize the lead from the soil into a solution by one or more of the following means:

☐ Changes in pH (by addition of an acid solution)

☐ Changes in system ionic strength (by addition of a suitable salt)

☐ Changes in redox potential (by addition of a suitable reducing agent)

☐ Formation of complexes (by addition of a ligand such as ethylenediaminetetraacetic acid [EDTA]).

Soil washing was first used in the Netherlands in the early 1980s and is widely used in Europe (Valenti, 1992). Soil washing starts with physical separation techniques to separate the coarse from the fine particles. The coarse fraction may be subjected to density separation to remove particulate metals. The fine fraction is mixed with a suitable wash solution to remove the lead bound to the soil. The coarse soil may or may not need washing depending on the amount of leachable lead associated with this fraction.

2.3.1 Acid Leaching and Contaminant Chemistry

Acid leaching helps to mobilize much of the fine particulate and soil-bound lead into solution by lowering the pH of the wash solution. Lowering the pH increases the supply of H+ ions, which are consumed in a multitude of reactions that increase soluble lead concentrations. Figure 2-6 shows how the solubility of lead compounds changes with pH. Except for lead sulfate (PbS), the solubility of the other compounds increases with decreasing pH.
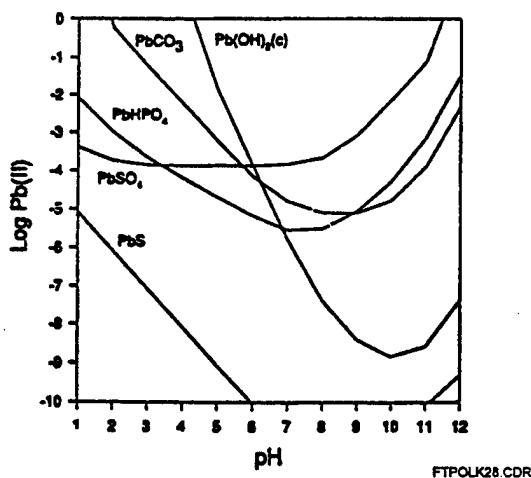
Figure 2-6. Pb Solubility Diagram: Calculations Made Assuming Solid Phase Always to be Present, with Total Chemical Component Concentrations [e.g., Pb$_T$, (SO$_4$)$_T$, (PO$_4$)$_T$, C$_T$] Varying Depending on Amount of Solid Phase that was Dissolved (van Benschoten, 1997)

Acetic acid and hydrochloric acid have been commonly used with lead contamination because both acids produce water-soluble salts. Acetic acid is weak and is expected to be effective at some sites where lead is mostly in the form of carbonate minerals (cerussite, hydrocerrusite, etc.). At small-arms range sites, lead carbonates may be formed from the weathering of elemental lead in the presence of native soil alkalinity (bicarbonates and carbonates). Other forms of lead, such as PbO_2 and lead sulfates, are more recalcitrant to solubilization by acetic acid. Lead sulfates are more common at lead acid battery sites. When lead sulfates or phosphates are present, the pH would have to be lowered to well below 3 to solubilize the lead. Extremely high molar concentrations of acetic acid (> 0.1M) would be required to attain a pH below 3, because of its low dissociation constant. As the concentration of acetic acid is increased, the percent dissociated decreases. In previous studies (Wozniak and Huang, 1982), pH 2 appears to be a threshold for lead and copper solubilization, whereas zinc was more easily solubilized, even at pH 3.

In general, solubilization rates were found to be dependent on pH, liquid:solid ratio, type of metal, and contact time. Of these parameters, pH and liquid:solid ratio are the limiting factors for a given metal. The pH determines the equilibrium solubility (concentration) achievable, and the liquid:solid ratio determines the total mass of metal removed. As far as contact time is concerned, in most cases, solubilization reaches a maximum in a relatively short time and then levels off (Wozniak and Huang, 1982), as long as relatively coarse metal particulates are absent. Metallic lead dissolves very slowly and, therefore, physical separation is desirable before leaching at small-arms range sites. A contact time between 10 to 60 minutes should be economically acceptable for a field leaching operation of the type conceived for small-arms ranges.

A stronger acid, such as hydrochloric or nitric acid, is more economical when the lead species requires much lower pH. A 0.1M solution of HCl, for example, has a pH of 1 and is more aggressive. Nitric acid may generate toxic oxides of nitrogen and is difficult to handle. Hydrochloric acid is therefore preferred. However, mineral acids such as hydrochloric acid are likely to be more corrosive on equipment and more aggressive on the native soil matrix.

2.3.2 Acid Leaching Process Configuration

Acid leaching is often done as a continuous process and involves at least four tanks as shown in Figure 2-7. In the leaching tank the acid solution is mixed with the contaminated soil to leach out the metals. The contact time between the leachant and soil can be set by designing the volume of the tank to achieve the required throughput rate. For a given volume of the tank, slowing down the throughput is the only way of achieving longer contact. Contact time requirements vary based on the type of soil and type of metal encountered. Small-arms range berms tend to be highly variable in terms of soil texture and contamination. Therefore, some degree of overdesign is advisable to maintain the desired throughput rate.

The soil slurry is pumped from the top of the leach tank to the clarifier, where the solids settle out and are discharged from the bottom. A flocculant may be added to enhance settling. The overflow from the clarifier is the leachate containing the solubilized metals. This overflow goes to a metal recovery tank, where the solubilized metals are recovered by electrowinning or, more commonly, by precipitation.

Acid ······

Flocculant ······

Regenerated Leachant

Precipitant ······
Flocculant ······

Unprocessed Solids

LEACHING TANK (0.5 to 1 hr.)

CLARIFIER (2 to 4 hrs.)

PRECIPITATION TANK (0.25 to 1 hr.)

CLARIFIER (2 to 4 hrs.)

Spent Leachant

Precipitate Sludge

PROCESSED SOLIDS DEWATERING

Processed Solids

FTPOLK31.CDR

——— Solids or Precipitate

– – – – Water or Dilute Slurry

············ Reagent
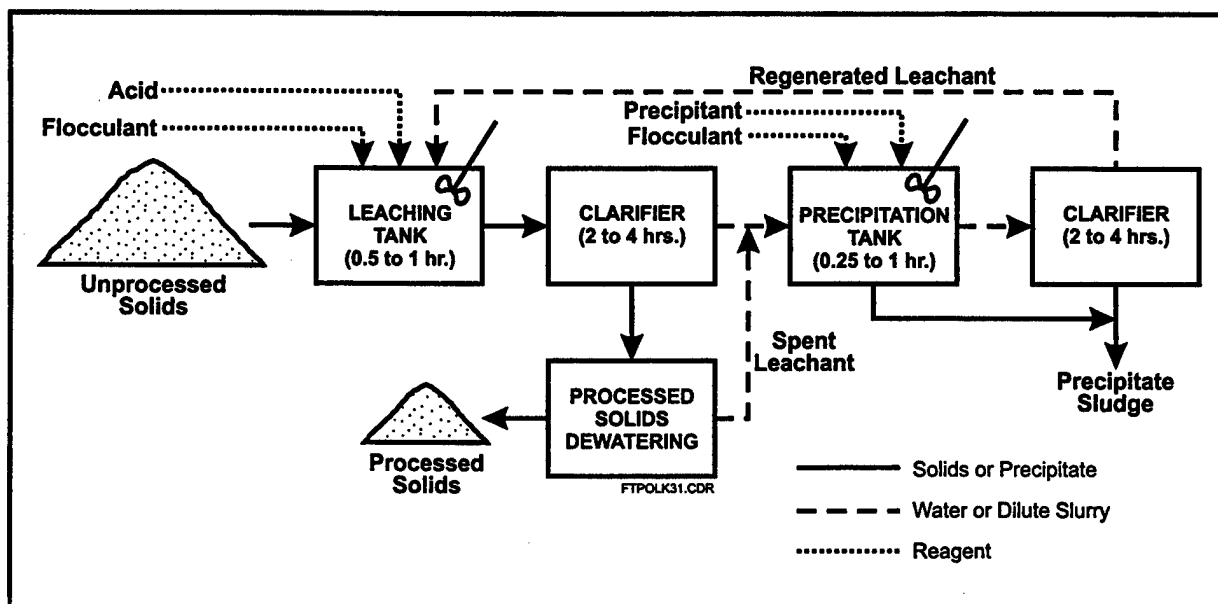
Figure 2-7. Continuous Leaching Process Flow

Precipitants used for metals recovery may include hydroxide, phosphate, carbonates, sulfate, sulfide, or other proprietary compounds (e.g., ThioRed®). The pH maintained in the precipitation process is an important determinant of the precipitation efficiency. The optimum pH is determined by the type of metal, type of precipitant, and presence of potential complexing agents, such as ammonia or EDTA. Figure 2-8 shows the theoretical precipitation curves for various metals as hydroxides (Lanouette, 1977). Lead, zinc, and chromium hydroxides have a pH optimum with hydroxide. As the pH is raised, solubility decreases up to a point. Beyond a certain pH, solubility starts increasing again. Therefore, pH control during precipitation is important. Also, if two or more metals are present in the solution, the optimum pH for each metal may be different from that for the same metals in individual solutions. A satisfactory pH, the use of polymers to aid in coagulation, the mixing and settling times needed, and the amenability of the precipitate to dewatering should be determined by bench-scale tests.

The treated leachant may then flow into a separate clarifier tank for settling of the precipitate. As shown in Figure 2-7, mixing of precipitant and coagulant with the leachant is fairly fast (15 to 60 min). Settling may require 2 to 4 hr at overflow rates of 300 to 700 gal/ft^2 of surface area per day (Lanouette, 1977). The respective tanks are sized accordingly. Some of the initial precipitate formed may be recirculated to the mixing tank, where the older precipitate particles provide a seed for new precipitate to grow on.

In the clarifier, the precipitate floc often settles down to form a sludge with only 1 to 2% solids. This sludge has to be dewatered before it is hauled away for disposal or recycling. The sludge can be dewatered in centrifuges, rotary vacuum filters, or plate-and-frame filters. Centrifuges
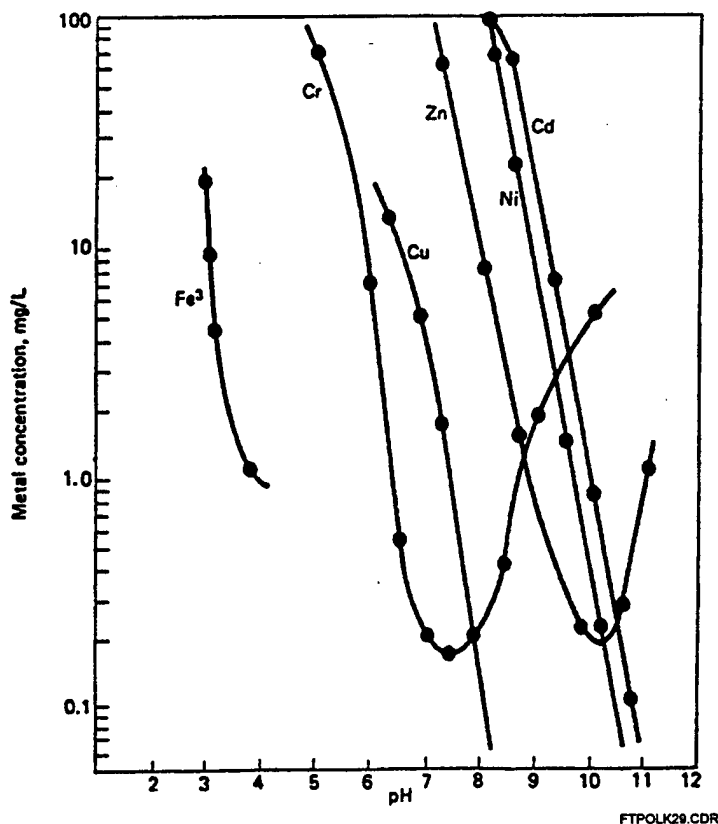
**Figure 2-8. Precipitation of Heavy Metals as Hydroxides
(adapted from Lanouette, 1977)**

require less floor space but may not dewater to the extent that the filters can. Plate-and-frame filters provide a drier cake and occupy less floor space, but require more operator attention than do rotary vacuum filters. A filter aid, such as diatomaceous earth, is recommended to prevent clogging of the filter cloth with fine precipitate particles. Bench tests can provide guidance on appropriate dewatering mechanisms.

The dewatered sludge can be sent to an off-site smelter for recycling if the lead content is high enough. The overflow from the clarifier is recycled back to the leach tank after being refortified with acid. An effective precipitation step assures the return of a good quality leachant containing low levels of soluble metals. This is important to maintain the performance of the entire acid leaching process.

2.4 ACETIC ACID VERSUS HYDROCHLORIC ACID
FOR SMALL-ARMS RANGE SOILS

Acetic acid (HOAc) and hydrochloric acid (HCl) have both been found effective for removing lead from contaminated soils. However, the efficacy and soil degradation/environmental impact of these acids vary with soil type and lead contaminant chemical forms. Although equimolar solutions of HOAc and HCl have the same neutralizing power, for example, as might be used to

neutralize caustic solutions between pH 6 and 8, these acids perform very differently in leaching metals in the pH <7 region due to their markedly different acid and buffering activity, metal complexing characteristics, and metal oxidation catalysis capability. Generally speaking, HCl is an aggressive leachant that is a corrosive and low-cost acid,-whereas HOAc is selective and far less corrosive but significantly higher in cost relative to HCl. In addition, HOAc may be more environmentally benign in the sense that it does not change the soil structure as does HCl.

2.4.1 Acid Activity Effect on Leaching Rate

Acid strength can be understood as the product of total acid concentration and hydrogen ion "activity," i.e., the fraction of the available hydrogen ion that is not already strongly bonded to something other than water (as "free" hydrogen or as H^+ dissolved in water). Bonded H^+ is not available to directly attack lead compounds (see below) to leach the Pb^{2+} contained in them, or to exchange with Pb^{2+} ions held by soil ion exchange sites. Therefore an acid, HX, dissociates partially when added to water to produce free H^+ according to the following reversible reaction.

$$HX \xleftrightarrow{K_a} H^+ + X^- \qquad \text{2-1}$$

HCl (where $X^- = Cl^-$) and HOAc (where $X^- = OAc^-$, or acetate) differ enormously (more than five orders of magnitude) in the extent to which Reaction 2-1 occurs in solutions of these acids. For example, when these acids are added to water and the resultant pH is <3.5, for HOAc the solution is composed of >90% neutral (un-ionized) HOAc molecules (Reaction 1 lies far to the left for HOAc). For HCl this same condition results in 100% of the HCl to be in ionized form, H^+ and Cl^- (that is, Reaction 1 lies far to the right for HCl). Therefore K_a (= $[H^+][X^-]/[HX]$, where [ ] = concentration) is very large (essentially infinite, i.e., HCl molecules are never present to a measurable extent in the pH >0 range). For HOAc, however, K_a is very small, $10^{-4.56}$ M (Martell and Smith, 1974), indicating that at about pH 4.5, 50% of the HOAc is present as the neutral molecule. Equation 2-2 can be used to estimate how the ratio of ionized to un-ionized concentrations of HOAc, $[OAc^-]/[HOAc]$, varies with pH.

$$\log\left(\frac{[OAc^-]}{[HOAc]}\right) = \log K_a + pH = pH - 4.56 \qquad \text{2-2}$$

The exponential relationship between the $[OAc^-]/[HOAc]$ ratio and pH (Equation 2-2) results in major changes in the fraction of HOAc ionized near where pH ~ $-\log K_a$ (or "pK_a"). Using Equation 2-2, these percentages are listed in Table 2-5.

On the other hand, HCl is 100% ionized over this same pH range. Note that as the $[H^+]$ is increased (decreasing pH), the fraction of acetic acid in ionized form becomes extremely small. Also note that the $[H^+]$ achievable from HOAc is always small. This last result is better shown by Table 2-6.

Table 2-5. Molecular and Ionized Forms of Acetic Acid Dependence on pH

pH	[OAc]/[HOAc] Calculated from Equation 2-2	% HOAc Ionized to OAc⁻	$[H^+]_{free}$ M
1.56	0.001	0.00099	2.8×10^{-2}
2.56	0.01	0.0099	2.8×10^{-3}
3.56	0.1	0.099	2.8×10^{-4}
4.56	1.0	50	2.8×10^{-5}
5.56	10	91	2.8×10^{-6}
6.56	100	99	2.8×10^{-7}

Table 2-6. Estimates of H⁺ Activity vs. Total Acetic Acid Concentrations Using Equation 2-2

Total Acetic Acid Concentration C_T^{HOAc} (M)	$[H^+]_{calc}$ (=[OAc⁻])	Calculated pH	[HOAc] M	% HOAc Ionized to H⁺ and OAc⁻
1	5.2×10^{-3}	2.28	0.99	0.53
0.1	1.6×10^{-3}	2.8	0.098	1.6
0.01	5.2×10^{-4}	3.3	0.0095	5.5
0.001	1.5×10^{-4}	3.8	0.00085	15
0.0001	3.6×10^{-5}	4.4	6.4×10^{-5}	36

From Table 2-6, it is clear that to achieve high percent dissociation of acetic acid into H⁺ ions, e.g., ≥15%, only very dilute acetic acid concentrations can be used (≥0.001 M). However, at these high dilutions the acidity is very low (pH ≥3.8). Therefore, optimum acid activity (H⁺ activity with minimum total HOAc concentration) will occur around pH 3.3 to 3.8. HCl has no such limit. HCl solutions will increase in acid activity with increasing total HCl concentration to the HCl solubility limit, about 12M or 37%.

2.4.2 Relative Lead Ion Complexation (Solubilization) Chemistry of Acetic and Hydrochloric Acids

Complexation reactions tend to solubilize metal ions in water. Both acetate and chloride ions display Pb^{2+} complexation capability but with much different strength and pH dependencies. Although chloride and acetate salts of most metals are highly water soluble, this does not always occur with complete dissociation of the ions of the salt by the water. Lead(II) is in fact a good example of this phenomenon where the anion associates (bonds to) the metal ion while in solution. These reactions are given in Table 2-7.

Table 2-7. Complexation Reactions Involving Lead

| Total Complexation Reaction | Symbol | Formation/Solubility Constants | |
		log K (OAc⁻)	log K (Cl⁻)
$Pb^{2+} + X^- \rightleftharpoons PbX^+$...(2-3)	K_1	2.15	0.90
$Pb^{2+} + 2X^- \rightleftharpoons PbX_2^0$...(2-4)	B_2	3.5	1.3
$Pb^{2+} + 3X^- \rightleftharpoons PbX_3^-$...(2-5)	B_3	3.18	1.4
$Pb^{2+} + 4X^- \rightleftharpoons PbX_4^{2-}$...(2-6)	B_4	3.4	0.7
$PbX_{2(s)} \rightleftharpoons Pb^{2+} + 2X^-$...(2-7)	K_{sp}	Very soluble	-5.0

Note: Interestingly, dialkyl lead(III) also forms mono and bischloro complexes with formation constants similar to those given in Table 2-7.

Although the formation constants for OAc are some 10 to 100 times those of Cl for Pb, the reactions 2-3 to 2-7 in Table 2-7 depend on the concentration of free anion, X, such that comparisons need to be made carefully (see below).

2.4.2.1 Acetic Acid

For HOAc the anion concentration can be very low in the pH 3.5 range (Tables 1 and 2). Note also that $Pb(OAc)_2$ is very water soluble, but $PbCl_2$ is not (Reaction 2-7). Therefore in the pH range of about 3.0 to 4.5, acetic acid provides both significant levels of available acidity (as HOAc) and some amount of complexing anion (OAc⁻), which combine to help solubilize lead from lead compounds in the soil. At higher pHs much less HOAc exists, and lead dissolution reactions that involve carbonates, bicarbonates, hydroxide, oxide, or organic acids are not leached as rapidly, e.g., Reaction 2-8.

$$PbCO_{3(s)} + 2HOAc \xrightarrow{\text{pH 3.5-4.5}} Pb(OAc)_2 + H_2O + CO_2 \uparrow \qquad (2-8)$$
$$\text{Soluble complex}$$
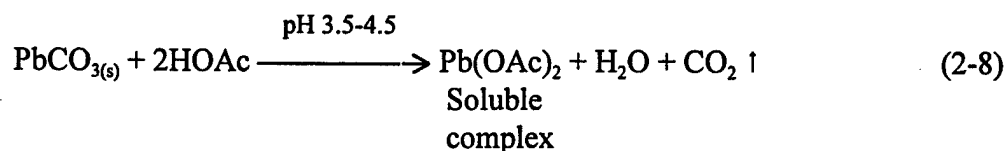
Critically, acetic acid can supply sufficient acid capacity (~0.01M) while still maintaining the pH window needed for Pb^{2+} solubilization by complexation (Table 2-6, columns 2 and 3).

Interestingly, if acidity is not needed to release the lead from the soil, and if it is not present as a highly insoluble, low-pH-sensitive salt (e.g., $PbSO_4$), lead complexation to form soluble acetate complexes can occur up to pH 7 to 8, above which hydroxide precipitation (to pH ~10 to 11) occurs, followed by redissolution by anionic hydroxide complexes at still higher pHs (pH 11 to 14) (Kragten, 1978).

2.4.2.2 Hydrochloric Acid

In comparison, HCl provides Cl⁻ which forms much weaker Pb^{2+} complexes, and $PbCl_{2(s)}$ solubility is only a little more than $PbSO_4$ solubility. HOAc is mostly present as neutral molecules in

the 3.0 to 4.5 pH range (Table 2-6); HCl is 100% dissociated over this same pH range. There-fore, a 0.01M HCl solution would have a pH of ~2, resulting in nonselective and substantial dissolution of many more materials. Also, the low solubility limits the total dissolved lead concentration to ~200 ppm, requiring large aqueous leach-phase (lixivient) volumes for heavily contaminated soils. Higher Cl levels would depress this solubility still further unless sufficient [Cl$^-$] exists to promote the formation of soluble complex species, remembering that even 0.1M Cl$^-$ increases Pb solubility only to a small extent.

Therefore HCl at mild pH ($\geq$3) (and therefore dilute conditions such as might be found with acid rain) has too little acid capacity ($\leq 10^{-3}$ M) to dissolve much Pb^{2+} from large acid-demanding solids such as lead carbonates (unless the pH is maintained with concentrated HCl additions). Therefore, relatively speaking, HCl has very little pH buffering or acid activity at pHs $\geq$3. The associated chloride from HCl does too little by complexation to promote Pb^{2+} solubilization and in fact could actually retard lead leaching through formation of low-soluble PbCl$_2$ particles. As PbCO$_3$ is more insoluble than lead(II) hydroxides and oxides (Kragten, 1978), this compound can play a dominant role in soils exposed to environmental conditions. The acetate complexes of Pb(II) are sufficiently strong to compete with carbonate and therefore mobilize Pb(II) in the pH 3 to 9 regime. Ten times higher concentrations of chloride ion are needed to impart similar effects (Kragten, 1978). The chloride ion effects, however, extend down to the strong acid region, e.g., to pH 1, which was the operating range used successfully by Vendor 2 during the field demonstration.

2.4.3 Aggressive Leach Conditions

Certain forms of Pb^{2+} ion are highly insoluble because the matrix in which the Pb^{2+} ion is trapped is highly insoluble. Ferrites, iron(III) hydroxides, and aluminum hydroxides, manganese dioxide (MnO$_2$), and PbSO$_4$ are such examples. The hydroxides and oxides can be amorphous or crystalline. Amorphous materials normally dissolve at faster rates due to larger surface areas, water content, and larger interatomic spacings (weaker bonds). The high stability of these materials results in slow rates of dissolution by acid. What is more, ferric hydroxide is already essentially completely precipitated by pH 2.5 (Baes and Mesmer, 1986). Hence, high H$^+$ activity is often required for rapid dissolution, and metals other than Pb also need to be dissolved to leach the Pb contaminants. Therefore, both high H$^+$ activity and high acid concentration are needed to achieve rapid leach rates. Normally only HCl can provide both of these conditions whereas HOAc can provide acid quantity but not the needed H$^+$ activity.

2.4.4 Catalysis of Oxidation

Chloride ion catalyzes electron transfer between metals and metal ions that are capable of oxidation reduction reactions. Chloride accomplishes this by bridging between two metal ions, thereby connecting their atomic orbitals for ease in electron transfer. Acetate ion is far less capable of electron transfer catalysis chemistry. Hence if Pb^0 is to be dissolved, or if ferrous iron enhanced dissolution of ferric and MnO solids is to be used, then Cl$^-$ addition may help promote dissolution rate. Increased acid activity promotes this reaction but it is not required. This same

catalysis phenomenon is responsible for the high corrosive action of chloride, especially HCl to carbon steel equipment.

2.5 FACTORS AFFECTING COST AND PERFORMANCE OF PHYSICAL SEPARATION AND ACID LEACHING

Table 2-8 summarizes the factors that affect the effectiveness, implementability, and costs for application of physical separation and acid leaching to remediation of small-arms ranges.

The proportion of fine materials in the soil is an important factor determining the effectiveness and cost of physical separation and acid leaching operations. The surface area is larger and settling velocity is lower for fine particles in comparison to larger particles of similar shape and density. Increased surface area increases the capacity of the soil for adsorbing contaminants, particularly if the fine particles are clay or humic materials. Lower settling velocity increases the residence time required to perform physical separations. Both factors tend to cause processing of fine particles to limit the throughput of a physical separation and acid leaching system. It is important to design equipment used for separation and leaching operations based on the measured content of fine particles in the soil. If the actual fine particle content is higher than the content measured in site characterization or treatability studies, the system capacity and performance will suffer. For example, a system with a clarifier designed to process 5 tons per hour of fine particles has a throughput of 50 tons per hour for soil containing 10% fines. If the soil contains 50% fines, the same system can process only 10 tons of soil per hour.

The chemical form of the metals strongly influences the selection of acid leaching chemical and operating conditions. The bullets enter the berm as elemental metals, but the actions of weather and chemicals in the soil alter the chemical state of the metals. Factors such as soil pH and the availability of anions (e.g., PO_4^{3-}, CO_3^{2-}, SO_4^{2-}, and NO_3^-) strongly affect the final equilibrium state and the rate of transformation. Therefore, design of the leaching and leachant regeneration system is site-specific.

The ability to conduct required unit operations with off-the-shelf equipment increases process reliability, flexibility, and ease of maintenance. Physical separation and acid leaching processes use well-established, mature unit operations. Commercial equipment is available in a wide range of sizes and construction materials from many manufacturers. Alternatively, an entire processing plant can be leased if the processing is contracted to a vendor.

Selection of the required throughput for the separation and leaching system is site specific. The system capacity should be sufficient to complete soil processing quickly so that the maintenance or remediation project does not interfere with the range mission at an active range or allows expeditious closeout of an inactive range. However, there are economic and performance limits on the maximum system size. High throughput requires large equipment that has higher capital costs and takes more time and expense to mobilize. If processing occurs very quickly, there will not be time to detect and adjust for processing problems such as inadequate metal removal. A processing rate sufficient to complete on-site processing in about 4 to 8 weeks typically is desirable.

Table 2-8. Factors Affecting Performance and Costs of Separation/Acid Leaching Technologies

Factor Influencing Selection	Basis	Data Needs
Equilibrium partitioning of contaminant between matrix and extraction fluid	Low partitioning of contaminant into the extraction fluid increases fluid volumes required to attain cleanup goal	• Equilibrium partitioning coefficient • Jar testing
Contaminant solubility	Soluble compounds can be removed by water flushing	Contaminant solubility as a function of pH and anion content
Complex waste mixture	Complex mixture increases difficulty in formulation of a suitable extraction fluid	• Contaminant composition • Priority pollutant analysis
Type and size of debris	Presence of debris increases pretreatment requirements	Waste composition
Particle-size distribution	• Particles >2 mm require pretreatment • Particles in the range from 0.063 to 2 mm are optimum for acid leaching • Acid leaching difficult with particles <0.063 mm (up to 20% clay may sometimes be tolerable)	• Sieve-size analysis of waste • Distribution of contaminants to various solid and liquid phases
Total metals concentration and distribution	Determine concentration targets or interfering constituents, pretreatment needs, and extraction fluid	Total metal concentration in various size fractions
Leachable metals concentration and distribution	Determine extractability of target constituents and posttreatment needs	Leachable metal concentration in various size fractions
Weathering	The amount of time that the metals are exposed to the environment affects their speciation	• Site history • Soil analysis
Extraction fluid characteristics	• Toxicity increases both health risks and regulatory compliance costs • Expensive or nonreusable fluid increases costs	• Fluid characterization • Jar testing • Pilot-scale testing
Waste buffering capacity and pH	High buffering capacity or pH increases acid consumption	Alkalinity
Presence of cyanides, sulfides, and fluorides	Determine potential for generating fumes at low pH	Waste composition
Matrix-specific surface area	High surface area increases sorption on soil	Specific surface area of matrix
Cation exchange capacity (CEC)	High CEC indicates the matrix has a high affinity for metal sorption	CEC of matrix
Clay content of waste	Clay increases sorption onto the waste matrix	Waste color, texture, and composition
Humic content of waste	Humic material increases sorption	Waste color, texture, and composition

Source: Adapted from U.S. EPA, 1995, EPA/540/R-95/512.

3. Site Description and Berm Soil Characteristics

3.1 SITE BACKGROUND

The two demonstrations were performed at the Army Base in Fort Polk, Louisiana, which is approximately 10 miles southeast of Leesville (Figure 3-1). Fort Polk was selected as the demonstration site because it has many active small-arms ranges with soils that are similar in character to soils at many of the DoD ranges (BDM, 1997). In addition, Fort Polk is environmentally proactive and has a good working relationship with the State of Louisiana.
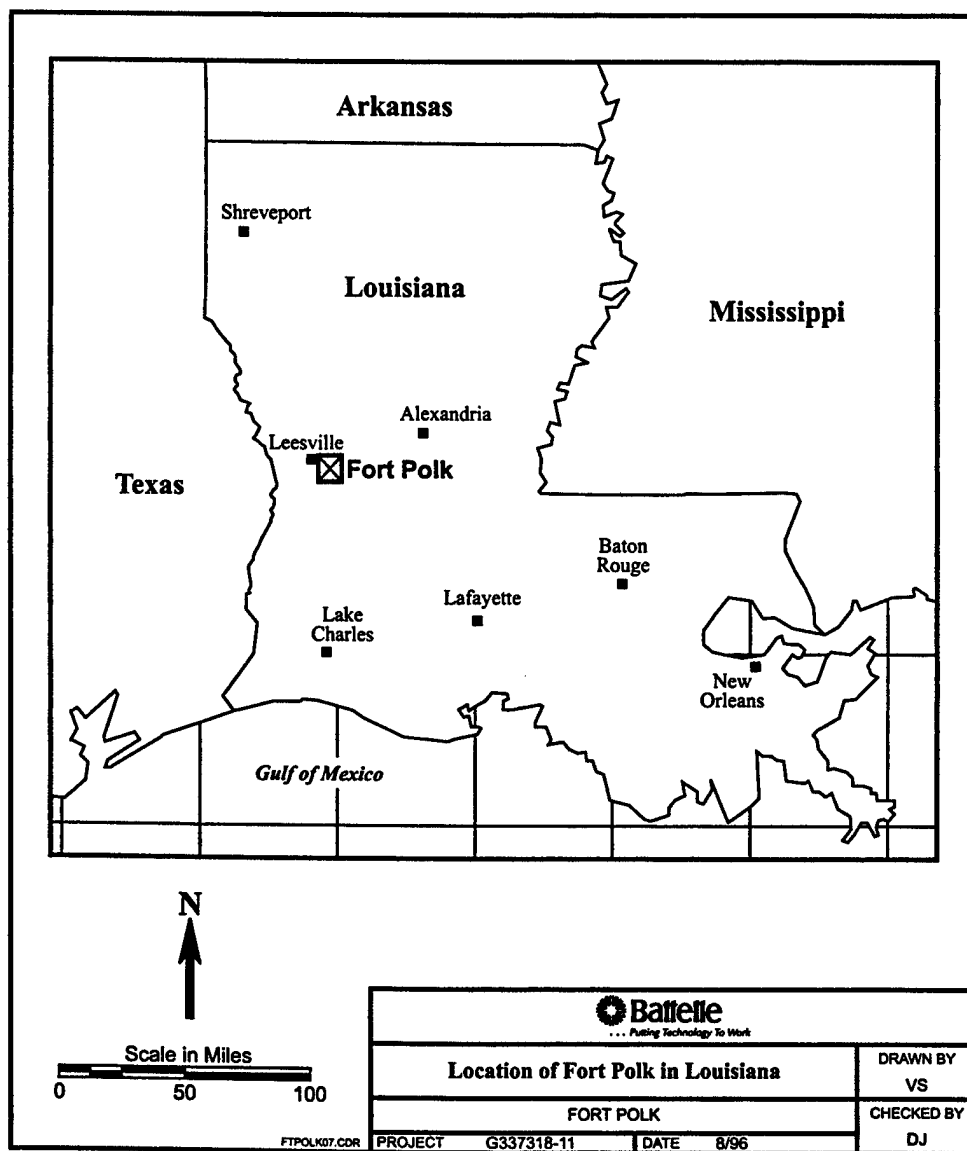
Figure 3-1. Location of Fort Polk, Louisiana

Range 5 is a 300-meter small-arms range that has principally supported M-1, M-14, and M-16 rifle training. Range 5 was selected for range maintenance because it is an active range, has a large amount of contaminated berm soils available, and was near to the proposed demonstration site (approximately 2 miles). Because the range is active, the demonstration was not designated as a remediation activity but as a routine range maintenance involving the removal and recycling of the accumulated bullet fragments.

3.2 DEMONSTRATION FACILITY CHARACTERISTICS

The demonstration site was located within a large block parcel (Block 4700) bounded by Service Command Road and Texas Avenue (see Figure 3-2). The demonstration area already had a well-established crushed asphalt and compacted gravel parking lot. A new impervious asphalt pad with bermed shoulders was installed by BDM, Inc., the site support contractor for the processing plant and soil stockpiles (see Figures 3-3 and 3-4). Because many of the physical separation and leaching operations are wet processes, it was assumed that spills and leaks would occur during processing, and provisions were made to contain them. The pad was graded so that runoff from the pad could flow into a lined 30,000-gallon stormwater runoff pond at the lower end of the pad. The pond also provided post-secondary containment for spills and leaks from the process plant. A valved sanitary pipe was installed to periodically drain the water from the containment pond into a sanitary sewer that leads to the Fort Polk sewage treatment plant or allow it to be pumped to a tanker truck and hauled off to a hazardous waste treatment facility, if required. Power was supplied to the vendor's plant through three transformers, each with 300 kVA capacity. Additional power for sample preparation equipment was arranged by Battelle through the rental of a 100-kW diesel generator. The vendors rented additional diesel generators for part of their power requirement. Process water was provided by channeling the base water supply through a backflow prevention tank.

A locked office/laboratory trailer was set up to house sample preparation/storage equipment, the x-ray fluorescence (XRF) analyzer, data storage/retrieval (laptop computer), telephone, restroom, and office desks. A small covered area outside the trailer was available to protect larger sample preparation equipment and the sampling personnel from the weather. The entire demonstration site was enclosed by a 6-ft-high chain link fence.

3.3 BERM CHARACTERISTICS

Figure 3-5 shows that Range 5 consists of three berms about 580 feet in length. Berm 1 has the lowest height of the berms at about 2 feet. Berm 2 has the highest crest and ranges from 5 to 8 feet. Berm 3 is about 5 feet in height. The site has been contaminated with lead from the firearms discharged during routine training exercises. Lead is present mostly in the berm soils (bullet pockets) located behind the firing targets. Additional soil with elevated levels of lead can be found between the berms. Background lead levels in Fort Polk soils appear to less than 50 ppm. In addition to lead, site characterization tests have shown that copper, antimony, and zinc are present in the site soils. Berm soil for the two demonstrations was excavated from Berm 3, and consisted of the top 18 inches of soil taken from the top of the berm to about 20 feet in front of the berm.
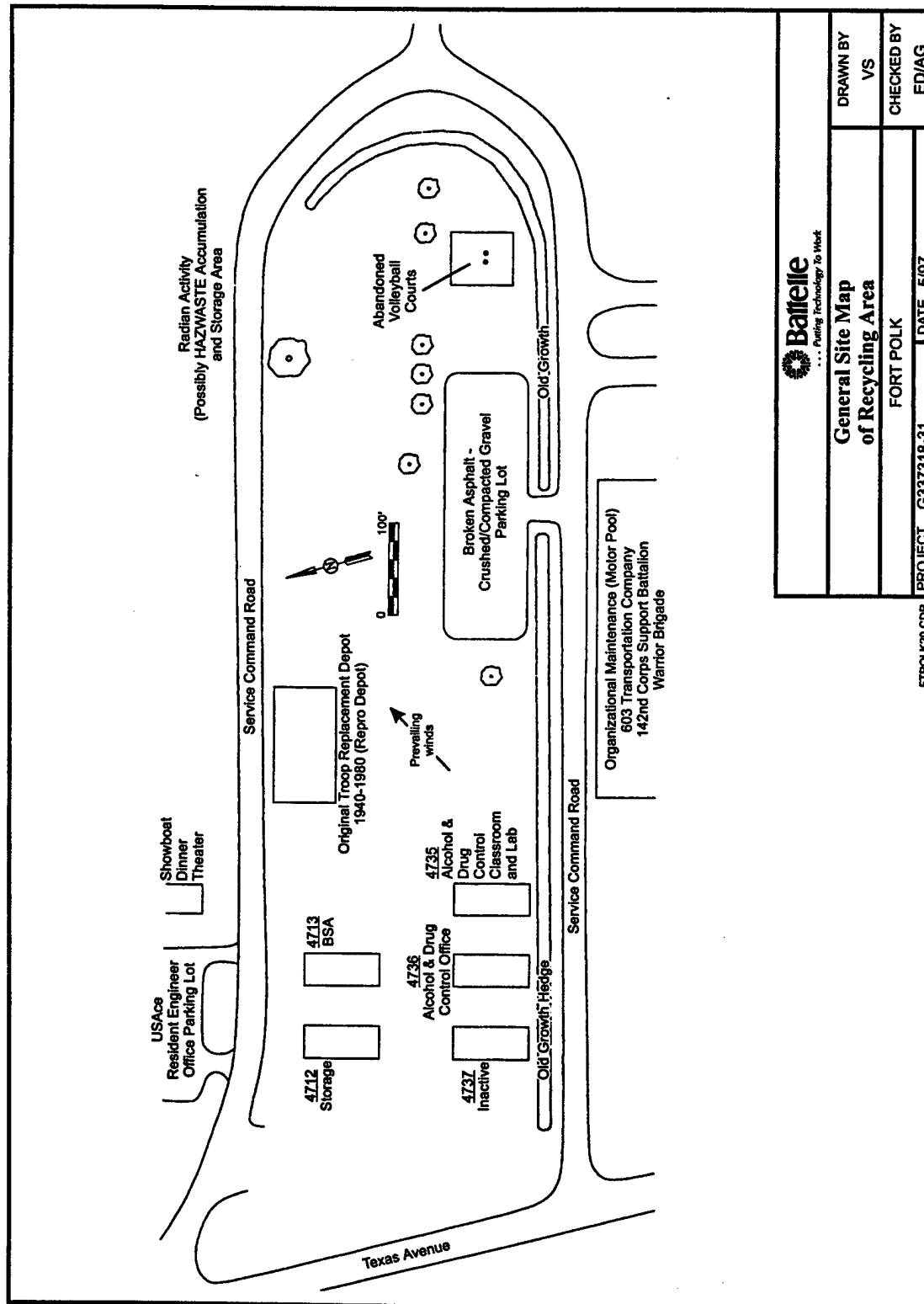
Figure 3-2. General Site Map Showing the Demonstration Site (Gravel Parking Lot)
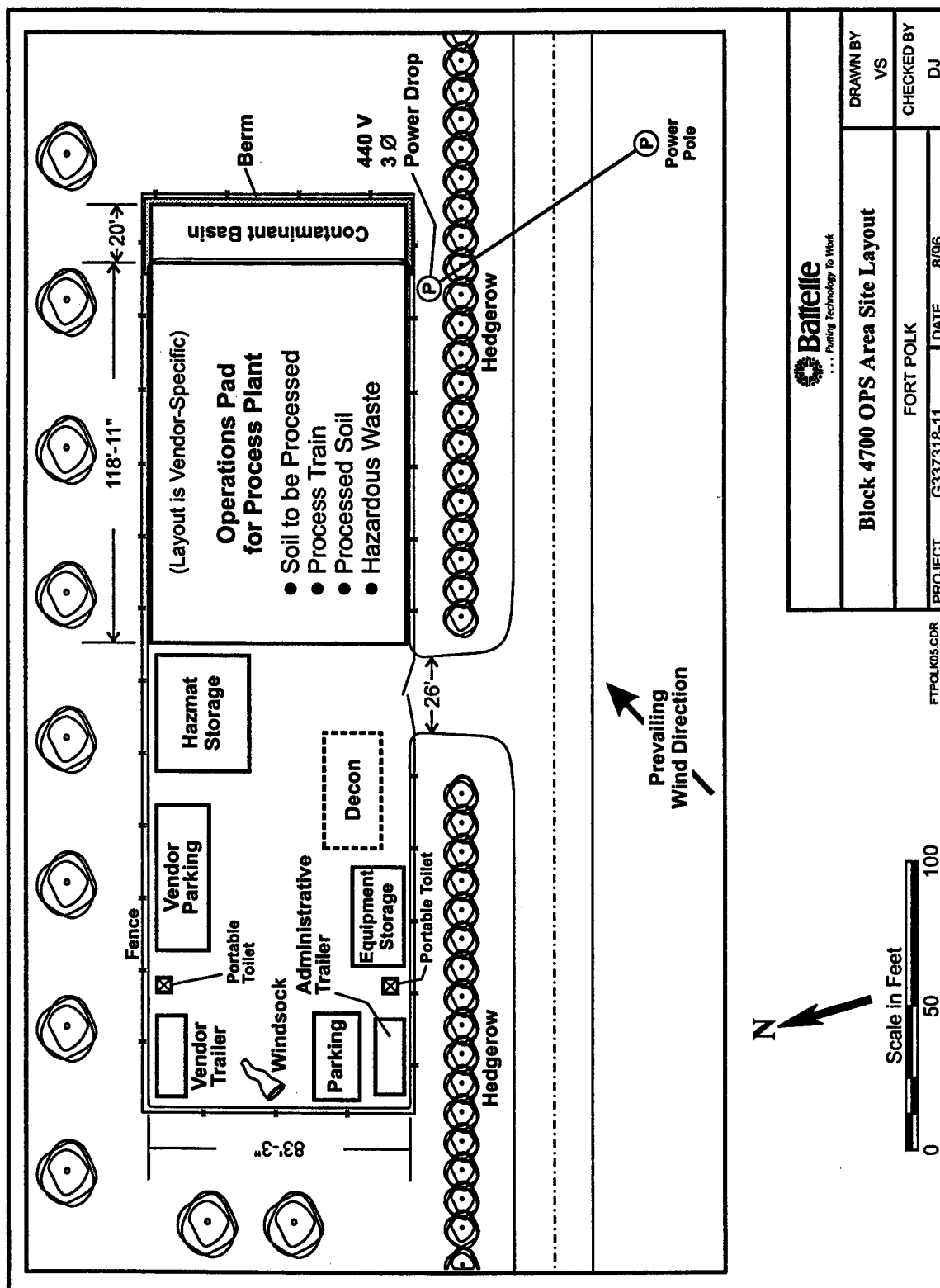
Figure 3-3. Layout of the Demonstration Site
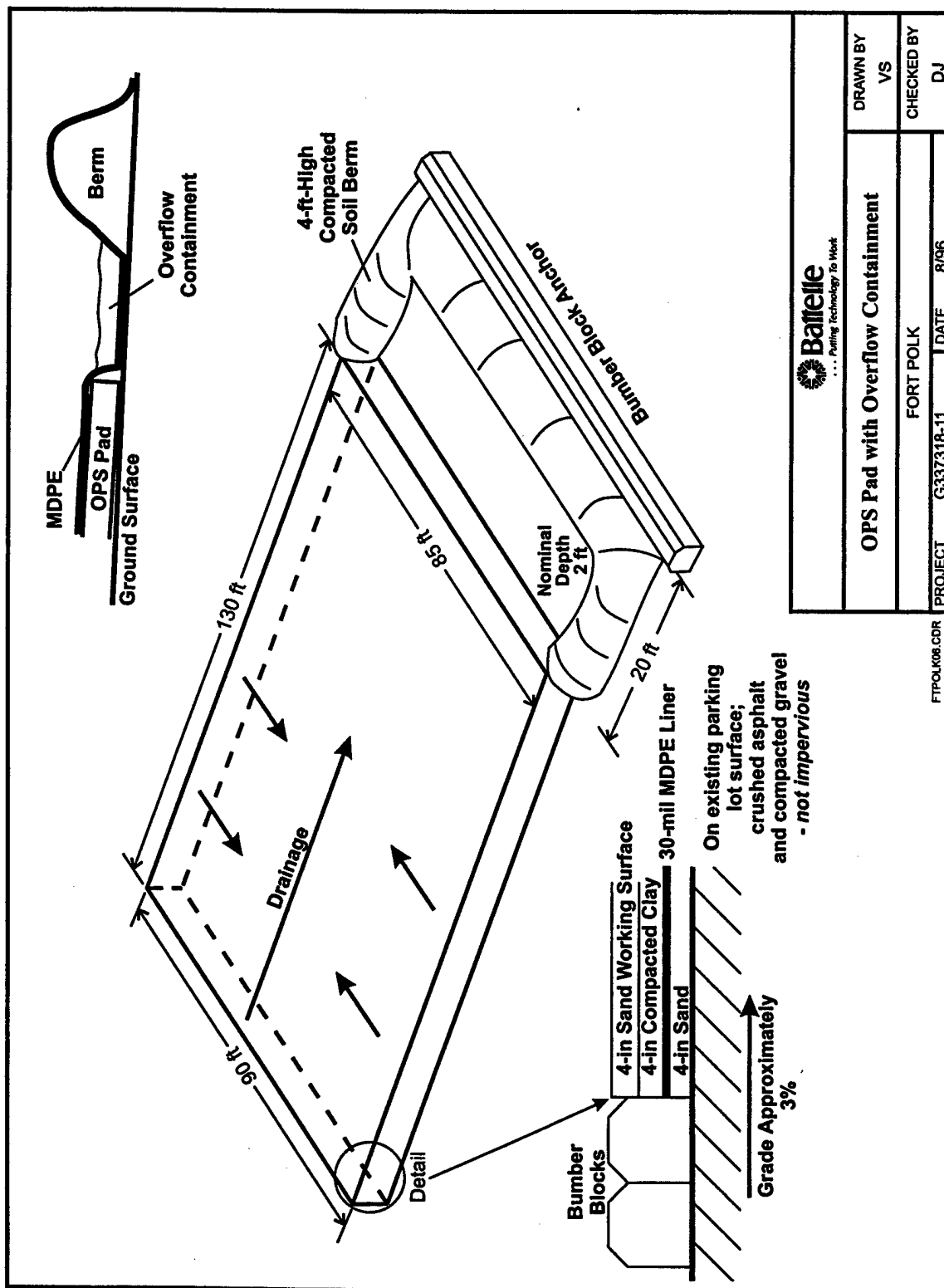
Figure 3-4. New Asphalt Pad with Overflow Containment Features Built by BDM for the Demonstration
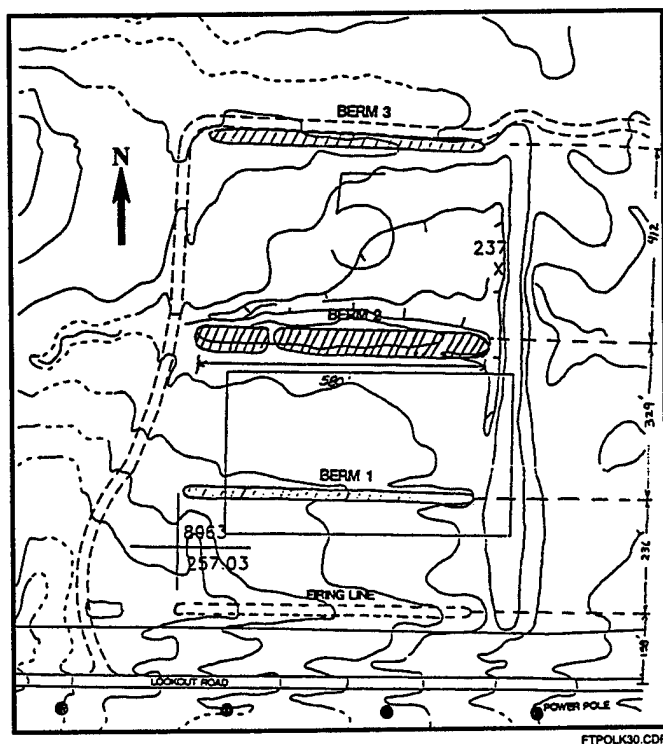
Figure 3-5. Range 5 Site Map

3.3.1 Characteristics of the Range Soil

The two most important characteristics of the range soil that should be known in order to design a physical separation/acid leaching system are the particle size distribution of the soil and the metal concentrations in the various size fractions.

The size distribution of the soil and particulate metals determines the type and size of separation and leaching equipment to be used. Wet sieve analyses reported by the two vendors from the samples of range soil sent to them for bench-scale treatability testing are shown in Table 3-1. Early range characterization conducted by the site on Berm 3 soil at Range 5 showed much higher gravel content (up to 34.3%) and much lower fines content (1.4 to 6.6%), based on pre-liminary site characterization information provided to the vendor by the site support contractor. One reason for this difference may be because the sample sizes in many of these cases were very small, given the site of the heavy metal particles in the matrix. Another reason could be because the fines tend to agglomerate and form balls of clay that are retained by the coarse screen during dry screening. Dry screening was a problem even in the field, and both vendors had to de-agglomerate the soil in attrition scrubbers before performing size separation or classification. In general, a wet sieve analysis is the preferred method for size analysis for characterization because any balls of clay tend to deagglomerate under the wet conditions. It also is the procedure recom-mended by ASTM (Method C117).

Table 3-1. Vendor Wet Sieve Analyses of the Raw Range Soil

Vendor 1 Screen Size	Vendor 1 % Distribution	Vendor 2 Screen Size	Vendor 2 % Distribution
> 1/4"	3.4	> 4 mesh	7.8
1/4" × 20 mesh	2.3	4 × 20 mesh	1.7
-	-	20 × 60 mesh	0.2
20 × 100 mesh	63.7	60 × 140 mesh	55.9
< 100 mesh	30.6	140 × 200 mesh	6.9
-	-	< 200 mesh	27.6

Vendor 1 - Acetic acid process.
Vendor 2 - Hydrochloric acid process.

Battelle conducted a particle size analysis on the raw range soil that was excavated and delivered to the demonstration site for processing. Table 3-2 shows the wet sieve analyses for two samples collected on different days from different raw soil piles. Although a checkerboard pattern of excavation was used to ensure uniform supply of raw soil in each pile delivered to the demonstration site, the fines fraction (−200 mesh) varied from 15.6% to 34.7% on different days. The amount of gravel (+4 mesh) also varied from 0 to 7.4%. This type of variability is a factor that should be taken into account in the design of any separation/ leaching plant. A lower-than-expected gravel fraction can slow down the screening process at the front end. A higher-than-expected fines fraction can slow down the leaching process. The plant has to have a flexible design with built-in excess capacity to handle this type of variability in the feed soil. Small-arms range berms may receive soil several times in their history from a variety of sources. Therefore, soil texture is most likely to be highly variable during processing.

Table 3-2. Battelle Wet Sieve Analyses of the Raw Range Soil

Battelle Screen Size	Battelle Field[a] % Distribution	Battelle Lab[b] Screen Size	Battelle Lab % Distribution	Battelle Lab[c] Screen Size	Battelle Lab % Distribution
> 3/8"	2.0	> 10 mesh	2.2	>3/8"	-
3/8" × 4 mesh	5.5	10 × 20 mesh	0.4	3/8" × 4 mesh	0.0
4 × 8 mesh	1.4	20 × 48 mesh	6.9	4 × 10 mesh	0.8
8 × 50 mesh	14.5	48 × 100 mesh	34.4	10 × 40 mesh	2.3
50 × 100 mesh	42.0	100 × 150 mesh	15.6	-	-
100 × 200 mesh	11.8	150 × 200 mesh	7.6	40 × 200 mesh	62.2
< 200 mesh	22.8	< 200 mesh	32.9	< 200 mesh	34.7

(a) Field distribution was averaged from the results of two analyses performed on September 16 and October 10, 1996.
(b) Hazen Research, Inc., Sample Date: December 5, 1996.
(c) DLZ Laboratories, Sample Date: November 14, 1996.

Following completion of the technology demonstration field activities, a more comprehensive evaluation of the Range 5 soil was conducted by the Battelle team on a more representative 30-gallon (318-lb dry weight) sample composited from a fresh pile of soil delivered to the demonstration site on December 5 (Hazen, 1997). The objectives of this range soil evaluation were to:

☐ Evaluate the soil particle size distribution from a large representative sample to verify previous results with smaller samples

☐ Evaluate the lead distribution in the various size fractions

☐ Examine range materials characteristics with respect to amenability to physical separation/acid leaching.

The procedures used in the Range 5 soil evaluation and the results are detailed in Appendix C. The results are described in Table 3-3 and Figure 3-6 and are summarized as follows:

☐ The raw sample soil contained 2.3% gravel (+10 mesh) and 32.9% fines (−200 mesh). The gravel fraction constituted 2.3% of the weight of the soil sample, but contained almost 80% of the total lead. Therefore, the majority of the lead is recoverable by relatively simple size or gravity separation techniques.

☐ The +10-mesh fraction was treated by hydrometallurgical and pyrometallurgical techniques to provide more separation information, as shown in Figure 3-3. The ferromagnetic fraction consisted of miscellaneous iron and one highly magnetic bullet. Very little lead was associated with this magnetic product. The nonmagnetic metals product contained 75% of the lead from the original soil sample. The nonmagnetic fraction was divided into a float

Table 3-3. Particle Size Analysis of the Raw Range Soil

Raw Soil (Total Weight = 142.5 kg)			Fraction Weight %		
Mesh size	Micron size	Fraction Weight (kg)	Retained (%)	Cumulative Passing (%)	Retained Weight (%)
Organics	N/A	0.08	0.1	99.9	0.1
+10	1,680	3.13	2.2	97.7	2.3
+14	1,190	0.22	0.2	97.6	2.4
+20	841	0.33	0.2	97.4	2.6
+28	595	0.94	0.7	96.7	3.3
+35	425	2.23	1.6	95.1	4.9
+48	297	6.54	4.6	90.6	9.4
+65	210	22.1	15.5	75.0	25.0
+100	149	26.9	18.9	56.2	43.8
+150	105	22.3	15.6	40.5	59.5
+200	74	10.8	7.6	32.9	67.1
−200	−74	46.9	32.9	N/A	N/A

N/A = Not applicable.
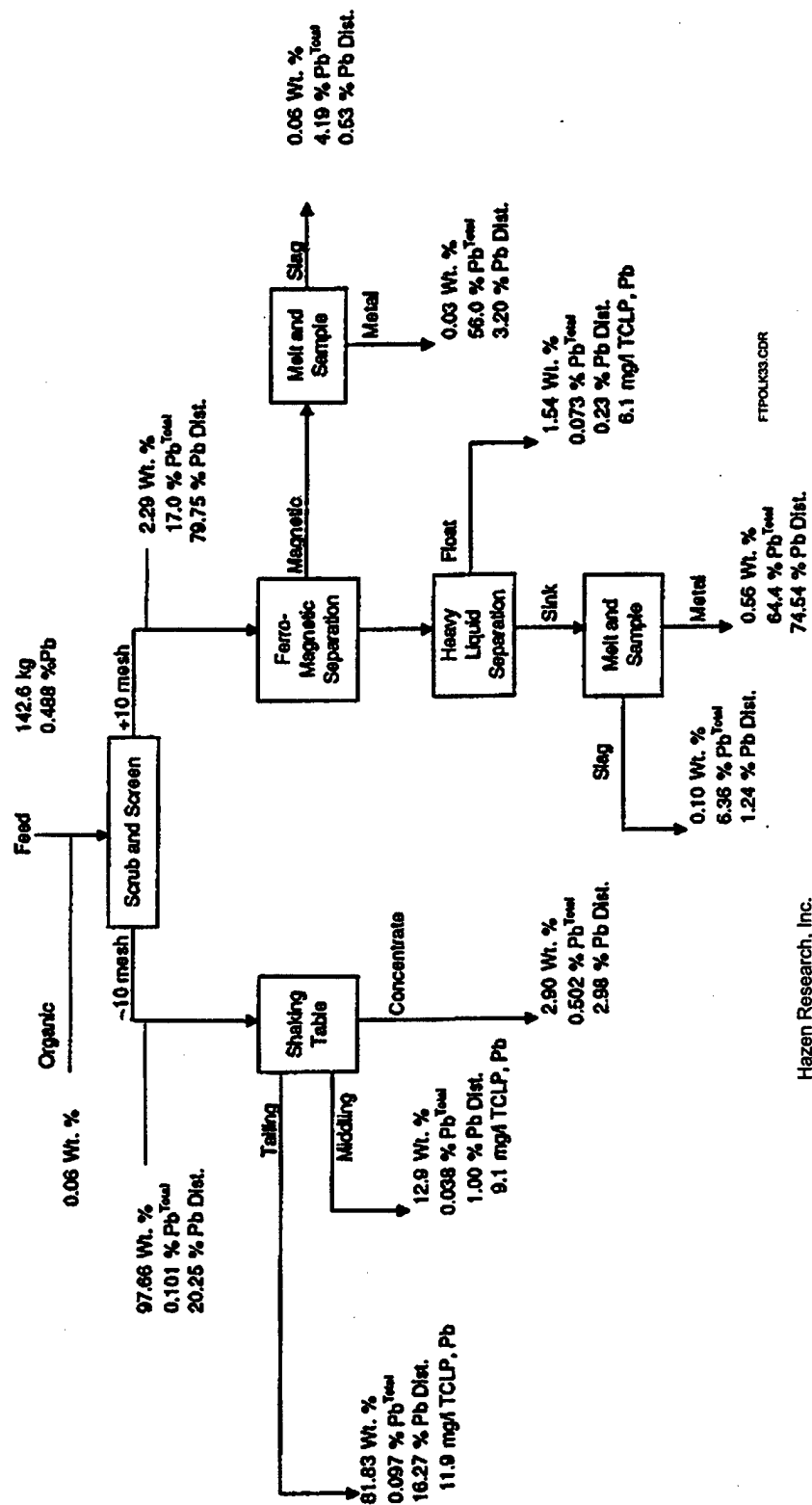
Figure 3-6. Lead Distribution Across Various Fractions of Raw Soil

(gravel) and sink (metals) product. Interestingly, the gravel (float) failed the TCLP criterion (5 mg/L) and would require further processing. Both vendors in this demonstration subjected the gravel material to some leaching to remove this small lead fraction. This was done by circulating the leachant solution through the physical separation circuit.

☐ The −10-mesh fraction (sands and fines) constituted 98% by weight of the soil sample, but contained only 20% of the total lead. The −10-mesh fraction was processed on a shaking table to see if the lead in this fraction was amenable to gravity separation. About 15% of the lead in the −10-mesh fraction (or about 3% of the lead in the original soil sample) was recovered in the table concentrate. The middlings and tailings both failed the TCLP criterion and would require further processing by leaching. The tailings contained 16% of the lead in the original soil sample, more than five times that in the concentrate. The usefulness of gravity separation (jig or shaking table) to the −10-mesh fraction is therefore somewhat uncertain. During the field demonstration, both vendors had trouble recovering heavy metals in their jig concentrates. It is not clear whether this happened because the metals were deposited elsewhere in the process (in the trough of the blade mill for Vendor 1, and on top of the jig bed for Vendor 2) or because the soil did not contain much lead that was amenable to gravity separation. The exact mode of occurrence of lead (particulate metal versus ionic lead species) in the fines was not determined in this characterization.

☐ An analysis of the water used for wet processing of the 30-gallon test sample showed a lead assay of less than 1 mg/L. This indicates that the pile of raw soil from the berm need not be covered to protect it from rain and infiltration into the ground, as long as the soil itself is contained and does not run off.

3.3.2 Contaminant Species

Because the demonstration was originally planned for Range 26, a lead speciation study (Honea, 1996) was conducted by the site for soil from this range rather than for the soil from Range 5, which was the range eventually addressed during this demonstration. The lead in the Range 26 soil appeared to be mostly in the form of carbonates (cerussite and hydrocerussite), with some laurionite [Pb(OH)Cl]. Bullet fragments were found to consist of a small antimony core surrounded by lead. Bullet jackets consisted of a copper-zinc alloy. Therefore, lead, copper, zinc, and antimony were the metals analyzed in the samples collected during the demonstration. In addition to lead, copper, zinc, and antimony are regulated in some states.

3.3.3 Other Soil Characteristics

Table 3-4 lists some additional characteristics of the soil measured by Battelle. The iron and manganese content of the soil is significant. Adsorption of heavy metals on iron and manganese oxides makes leaching more difficult, especially when a weak acid such as acetic acid is used. The heavy metal fraction bound to organic matter, represented here by the soil's TOC content, also is difficult to remove by leaching with a weak acid. In comparison, the exchangeable heavy metal fraction can be removed easily by both weak and strong acids (van Benschoten et al., 1997).

**Table 3-4. Other Small-Arms Range Raw Soil Characteristics
Measured by Battelle**

Parameter	Measured Value
Soil description	Silt-sand
Moisture content	8%
Total organic carbon	7,710 mg/kg
Iron	12,115 mg/kg
Manganese	50.7 mg/kg
Cation exchange capacity (CEC)	4.5 meq/100 g

3.3.4 Excavation and Homogenization of Range 5 Soil for Processing

Contaminated soil from Berm 3 at Range 5 was excavated for processing. The site support contractor used a checkerboard pattern of soil excavation in an attempt to provide both vendors with approximately the same type of raw soil for processing. The soil was excavated using a backhoe and placed near Range Road for easy access. The soil was placed to form a pile of approximately 150 tons for each batch and was covered with tarpaulins to prevent the soil from receiving excess moisture due to rainfall. The soil pile was routinely mixed and turned by the backhoe/front-end loader. From Range 5, the soil from the pile was loaded onto dump trucks of 30-ton capacity. The dump trucks transported the raw soil to the demonstration site (Building 4700) and dumped it on the asphalt pad to form a new raw soil pile. Raw soil was brought to the site only when necessary. During the Vendor 1 demonstration, raw soil was delivered to the site on four separate occasions. However, during the Vendor 2 demonstration, which progressed much faster, raw soil was delivered to the operations pad almost daily.

4. Objectives and Approach

The overall goal for this demonstration was to evaluate and document the technical and cost performance of two processes that use a combination of physical separation and leaching technologies to remove lead and other metals from small-arms range berm soils. Vendor 1 used acetic acid for leaching and Vendor 2 used hydrochloric acid. The goal was not to compare the two processes but to evaluate them independently for their potential application to different site conditions that may be encountered at different locations.

4.1 PERFORMANCE OBJECTIVES

The technical performance of the two processes and their application to small-arms ranges was evaluated through the following objectives:

☐ Ability of the continuous process to reach and maintain steady state at a rate of 5 to 10 tons/hr.

☐ Ability of the processes to generate a final treated material that contains less than 1,000 mg/kg (or 500 mg/kg for the hydrochloric acid process) of total lead

☐ A good faith attempt to generate a final processed material that meets TCLP limits for lead ($\leq$ 5 mg/L)

☐ Suitability of the physical characteristics (ease of handling, moisture content, and other visual observations) of the processed material for return to the range

☐ Acceptability of the concentrated lead fractions (metals concentrate stream and precipitate sludge) for off-site recycling; determination of the quantity and characteristics (hazardous/nonhazardous) of any nonrecyclable residuals generated during processing.

The cost-effectiveness of the two processes for small-arms range applications was evaluated through the following objectives:

☐ Estimation of the variable operations and maintenance (O&M) costs of the process, including labor, power, reagents, waste disposal, recycling, and monitoring (sampling and analysis)

☐ Estimation of the fixed (capital) costs for the remediation, including mobilization/demobilization, site preparation, environmental planning, and permitting

☐ Estimation of cost per unit mass (ton) of berm soil processed.

4.2 APPROACH AND ACTIVITIES

The following activities were undertaken to achieve the demonstration goals:

☐ Site Selection. USAEC and NFESC selected Fort Polk because of the availability of suitable berms and for its proactive environmental stance.

☐ Regulatory Review and Approval. The demonstration was accepted as a range maintenance activity. An environmental assessment of impacts was conducted by BDM, Inc., the site support contractor. Battelle worked with the base safety officer to obtain the necessary permits to use the x-ray fluorescence (XRF) unit at the site for lead screening. Fort Polk conducted a safety inspection of the site arrangements before processing began.

☐ Site Characterization. The site support contractor, BDM, did a preliminary characterization of the range soils and collected representative samples to send to the selected vendors for their bench-scale tests.

☐ Worldwide Search and Vendor Selection. BDM conducted the search and selection process that identified the two vendors who were contracted to demonstrate their processes. Vendor 1 was asked to use acetic acid leaching. Vendor 2 was asked to use hydrochloric acid leaching.

☐ Bench-Scale Tests. The two vendors selected conducted bench-scale tests on samples of berm soil provided by the site support contractor to aid in the design of the process plant.

☐ Site Preparation. The site support contractor, BDM, constructed the processing pad and arranged for the utilities supplied to the process plants.

☐ Technology Demonstration Plan Preparation. Battelle, under contract with NFESC, prepared a Technology Demonstration Plan for the independent evaluation of the technology and the demonstration activities. The plan was reviewed by NFESC, USAEC, BDM, and the two vendors, and was approved by ESTCP.

☐ Vendor Demonstrations. Between August and December 1996 at the Fort Polk site, Vendor 1 demonstrated the acetic acid process and Vendor 2 demonstrated the hydrochloric acid process.

☐ Site Verification. By collecting and analyzing soil samples from the demonstration site before and after the field activities, Battelle and BCM confirmed that no residual contamination was left behind.

☐ Reporting. The final report (this document) evaluating the two vendors' processes and their implementation was prepared by Battelle.

5. Description of the Process Plants Used at Fort Polk

This section describes the process plants used by the two vendors during the Fort Polk demonstration. Vendor 1 used an acetic acid-based process, whereas Vendor 2 used a hydrochloric acid-based process. Both vendors used similar types of physical separation and leaching equipment, although the configuration and sizes varied. The figures in this section show the process streams and locations that were sampled during the demonstration.

5.1 ACETIC ACID PROCESS

Figure 5-1 is a photographic overview of the plant used by Vendor 1. The plant utilized a physical separation module coupled with a leaching module that employed acetic acid as the leachant. The plant that was assembled at the site on September 3, 1996 is shown as Figure 5-2. The main plant consisted of three self-contained flatbed trailers mounted with processing equipment. In addition to the trailers, other significant equipment included an acid storage tank, three clarifying tanks, a feed hopper and conveyor, a processed soil belt conveyor, a vacuum belt filter, and two surge pumps. The plant was plumbed and system connections were made during the next few days. Additionally, the plant was filled with approximately 20,000 gallons of water and acetic acid to bring the operating pH at most locations to 3.2.

On September 7, 1996, some raw soil from Range 5 was brought to the operations pad and run through the system. After several adjustments and some effort to minimize the leaks from the system, the first significant processing was conducted on September 15 as a pilot run. Subsequently, other operational issues, such as a variable feed soil matrix, operating pH level, and soil dewatering, had to be addressed through more plant modifications. Figure 5-3 shows the final configuration of the plant that was demobilized on October 14, 1996. The following sub-sections describe in more detail the physical separation and acid leaching modules of the plant, and the changes that evolved.

5.1.1 Physical Separation Module

Figure 5-4 shows the final configuration of the physical separation module. Streams that were sampled for this evaluation are marked in this figure by alphabetical symbols. The physical separation module initially separated the coarse soil (+175 mesh) from the fine soil (−175 mesh). The coarse material was subjected to density separation to remove particulate metals. The fines were sent to the leaching module. Under ideal conditions, a pH of 3.5 was to be maintained in the washwater used in the wet physical separation process.

5.1.1.1 Size Separation

Raw soil from the stockpile was loaded into the feed hopper and fed to the attrition scrubber via the belt conveyor. The feed hopper used for this demonstration was equipped with a plate feeder. The conveyor was approximately 34 feet long, and was equipped with load cells for determining and recording feed rates and totals. The conveyor was also fitted with a magnetic separator that was used to separate nonmagnetic from magnetic material. During the demonstration, the feed hopper
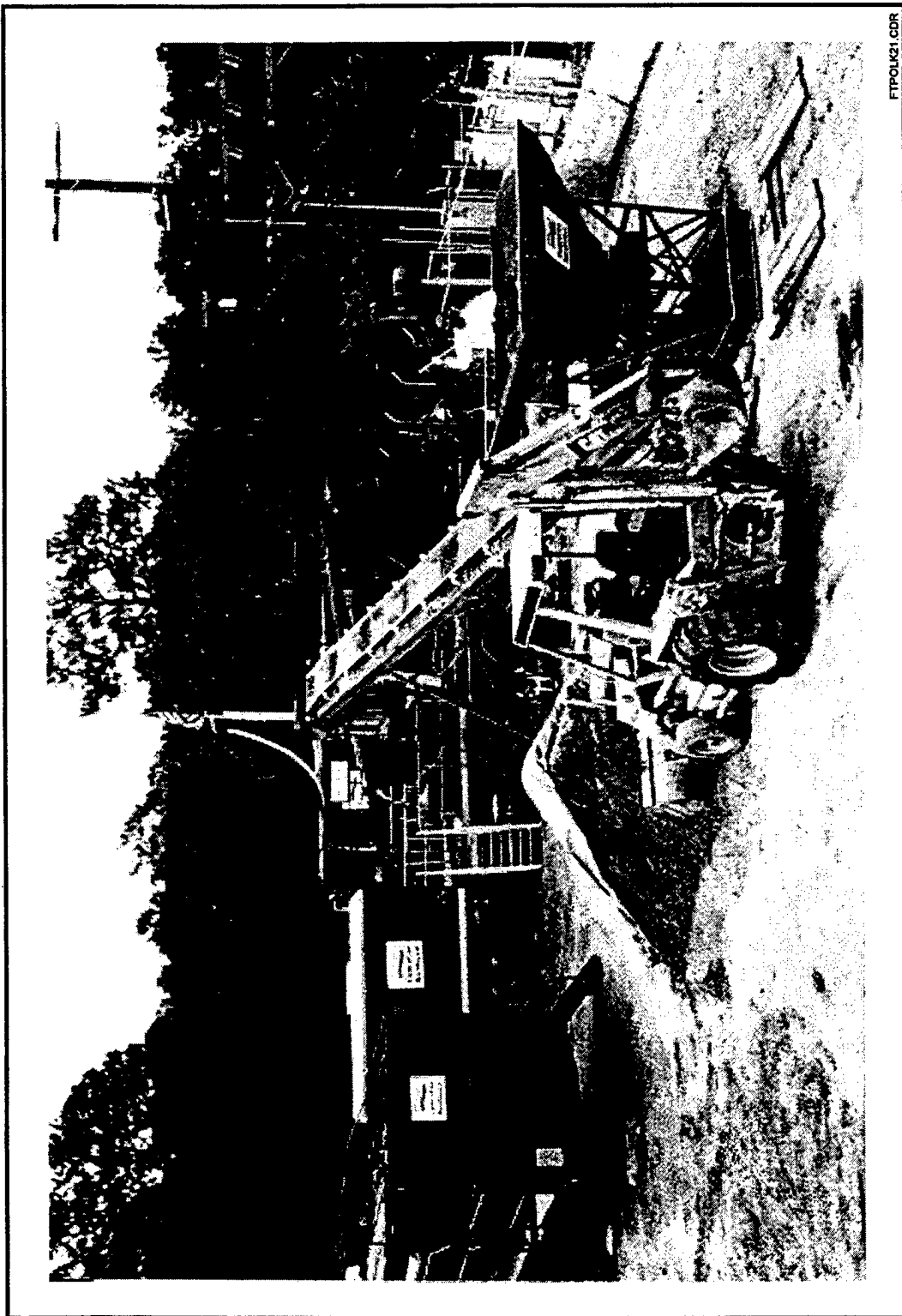
Figure 5-1. Overview of the Acetic Acid Process
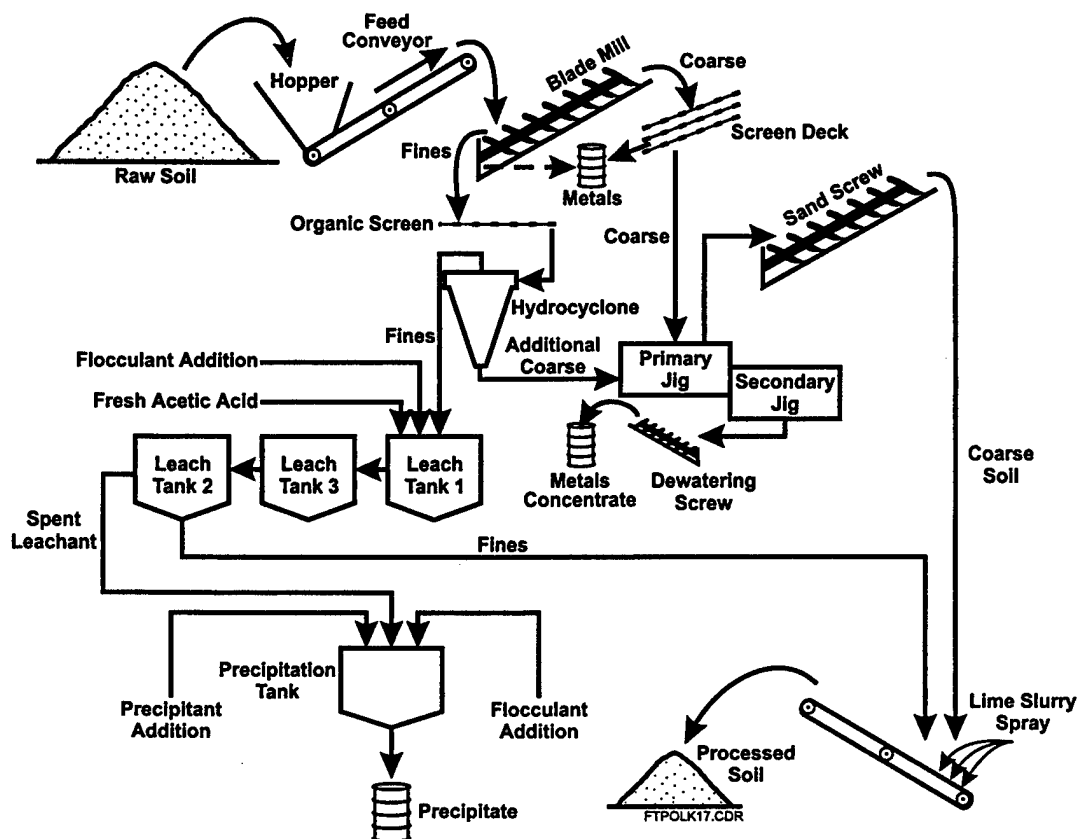
Figure 5-2. Initial Schematic of the Acetic Acid Process

often malfunctioned because of soil bridging, whereby the soil sticks to the sides of the hopper and is unable to drop onto the feeder. An operator had to manually knock soil from the walls of the hopper onto the plate feeder. The plate feeder itself failed on September 21. The vendor then used a backhoe to feed raw soil to the plant until September 25 when the plate feeder was modified.

Initially, a blade mill was employed to deagglomerate clay clumps through attrition. Initial processing revealed that the blade mill was not able to de-agglomerate the soil sufficiently. A dual-cell attrition scrubber was subsequently ordered and arrived on site on September 12. It was installed and effectively served to break up soil clumps and slurry the soil. The blade mill was retained and functioned as a mechanical classifier. The overflow stream from the blade mill passed through a ¼-inch screen set at a 45° angle to separate organic matter from the dilute slurry of fines. At Fort Polk, the separated organic matter had a higher lead concentration than the soil. However, during the course of the demonstration, the screen was placed flat across the hydrocyclone surge tank to stop process water from leaking from the screen deck onto the operations pad.

A triple-deck vibrating screen equipped with jet spray nozzles was attached to the discharge end of the blade mill. The screen decks had the following mesh sizes: ¾", ½", and ⅜". Because the raw soil fed to the process did not contain significant amounts of material greater than ⅜", the screen decks were only vibrated occasionally. Oversized gravel particles were retained by the
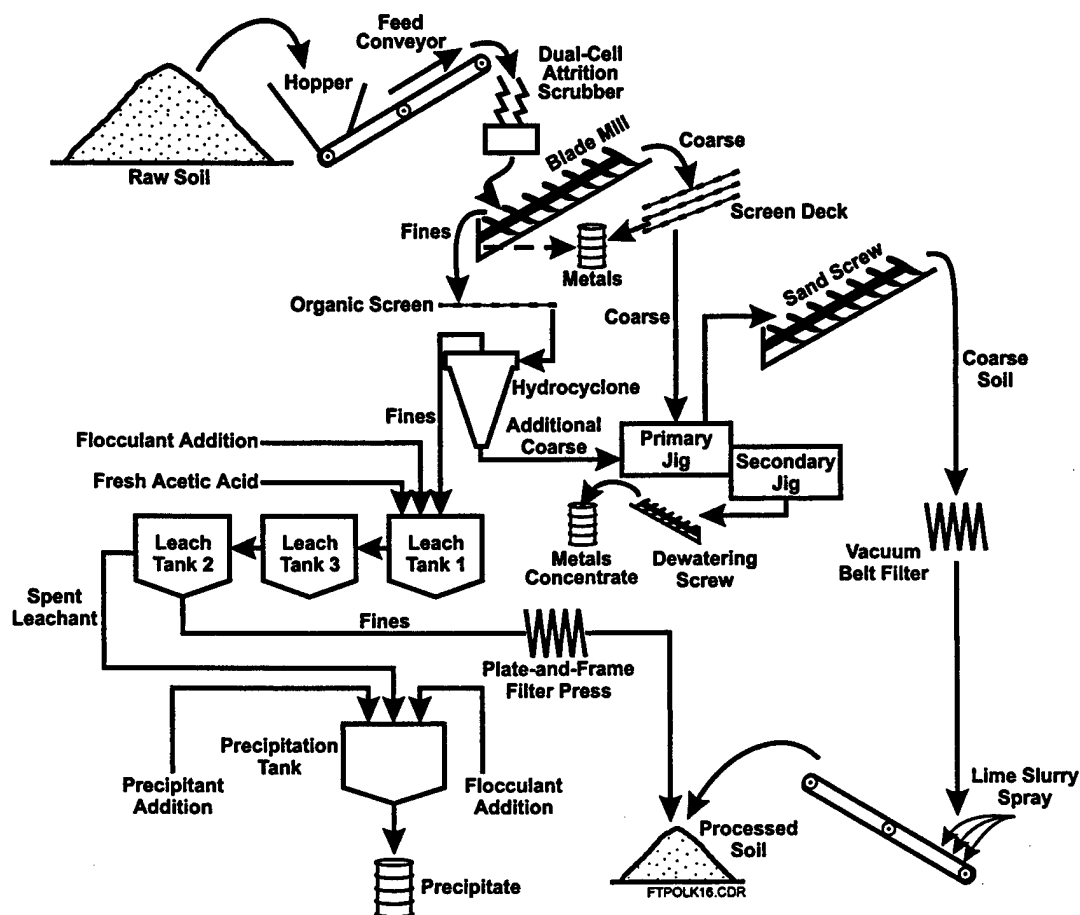
Figure 5-3. Final Schematic of the Acetic Acid Process

¾" screen deck and sent directly to the processed soil conveyor. The –¾" + ½" material was also spray-washed and sent to the processed soil conveyor. The –½" + ⅜" material was expected to contain a majority of the bullets and metal fragments from the feed soil. This material was diverted to 55-gallon drums for off-site recycling. However, most of the bullets and gravel in this fraction were found to have settled out in the bottom of the blade mill. These metal fragments were recovered from the blade mill during plant demobilization.

The undersize (–⅜") from the screen decks was pumped to the density separation units. A hydrocyclone was used to further separate the fines from the blade mill overflow before being pumped to the leaching module. Additional coarse particles (+175 mesh) were pumped from the hydrocyclone to the density separation portion of the plant.

5.1.1.2 Density Separation

Density separation for the plant consisted of primary and secondary jigs. Coarse soil greater than 175 mesh was fed to the density separation units. Each jig was designed to remove, as underflow, 10% of the material that was fed to it. The underflow from the primary jig fed the

Figure 5-4. Physical Separation Process (Acetic Acid Process)

BATTELLE *September 22, 1997*
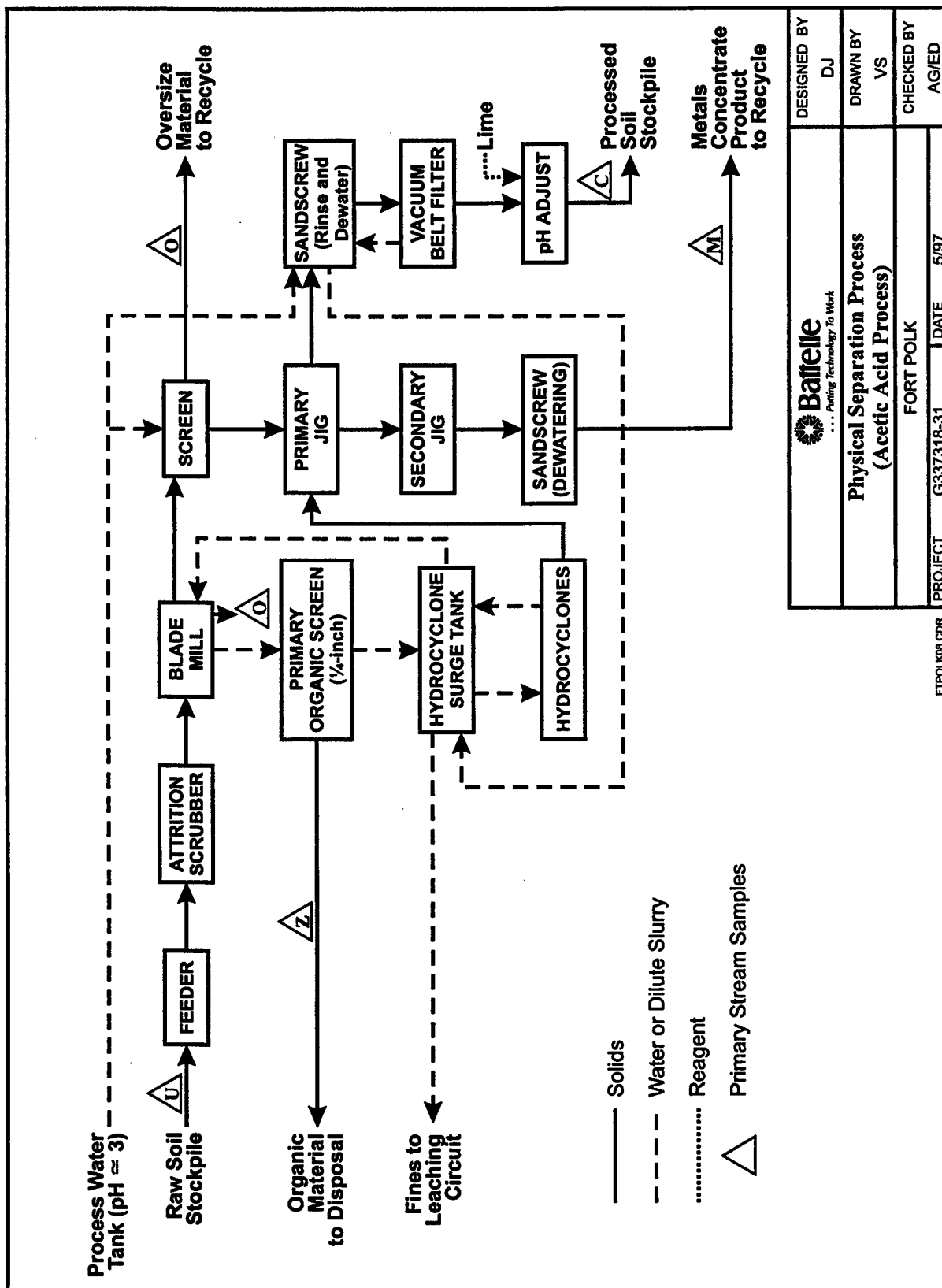
secondary jig, where 10% of that material was removed as underflow (probably metals) and pumped first to a small sand screw for dewatering and subsequently to 55-gallon drums for off-site recycling. The overflow from the secondary jig was returned to the primary jig in a closed-loop circuit to achieve a more efficient density separation. The overflow from the primary jig bed was pumped to a sand screw.

The sand screw, approximately 30 feet in length, was used to dewater the coarse processed soil. The dewatering capability of the original sand screw was improved by expanding its basin volume. The overflow from the sand screw basin was pumped back to the hydrocyclone surge tank. The soil discharged from the sand screw was sent to the processed soil conveyor, where an attempt was made to neutralize it with hydrated lime to a pH of 5.5. Eventually, the coarse material was further dewatered on a vacuum belt filter to solve the material-handling problems resulting from the wet processed soil.

5.1.2 Leaching Module

Figure 5-5 shows the leaching module utilized by Vendor 1. Although acetic acid solution (pH of 3.5) was used as washwater in the physical separation module, most of the leaching took place in three 2,000-gallon tanks equipped with bottom sweeps. Each tank had a controlled pH of 3.2. During bench-scale testing, Vendor 1 had concluded that effective acid washing of the Fort Polk soil could be achieved at this pH and a residence time of 200 minutes.

5.1.2.1 Acid Leaching of Fines

The dilute slurry of fines from the hydrocyclone surge tank was pumped directly to the first 2,000-gallon leach tank. In this tank, the fines were contacted with acetic acid solution at a pH of 3.2. The pH in the tanks was controlled by a constant-pressure transfer system that used pH meters and solenoid valves to automatically inject fresh acid into Tank 1 should the pH rise. Water was pumped into the tanks if the pH was lower than desired. The leaching system was semicountercurrent. By circulating leachant in this fashion, it was hoped that cleanup criteria could be achieved with regularity. It was also necessary to introduce polymeric flocculant into the leaching circuit to allow the solids to settle as a sludge in each tank. Flocculant was added by manual adjustment of variable-volume piston pumps. The solids traveled from Tank 1 to Tank 3 to Tank 2.

The sludge from the final leaching tank (Tank 2) initially was pumped to a vacuum belt filter operated at a vacuum of 20″ Hg. The wash solution removed from the processed fines was sent to the precipitation tank via a recirculation pump. During the demonstration it was determined that the vacuum belt filter was too small to dewater the mass of processed fines sent from the leaching circuit. Therefore, a plate-and-frame filter press was ordered and arrived on site on September 30, 1996. The filter press served as the fines-dewatering mechanism for the the next few days, until the filter cloths became clogged and had to be replaced. After the filter cloths had been changed, the plate-and-frame filter press was used for the duration of the demonstration. This press was able to reduce the moisture content in the fines to approximately 30%. As previously mentioned, the original vacuum belt filter was instead used for additional dewatering

Figure 5-5. Leaching Circuit (Acetic Acid Process)

of the processed coarse soil. The overall moisture content of the final processed soil (coarse and fine) was reduced to approximately 22%. The wash solution removed from the processed soil by the two filters was sent to the precipitation process via the same recirculation pump.

5.1.2.2 Precipitation Process

Effective precipitation of metals and regeneration of the acid leachant are essential to the overall performance of a leaching plant. The precipitation portion of the plant is shown in Figure 5-5. The precipitation process consisted of one 2,000-gallon tank with bottom sweep, one clarifying tank with baffles, two more clarifying tanks in series, and a proprietary chemical, ThioRed®, used as the precipitant. ThioRed® is a commercially available, biodegradable polythiocarbonate.

The ThioRed® precipitant was introduced into the precipitation tank along with a polymeric flocculant. The precipitate sludge was pumped into 55-gallon drums for recycling purposes. Precipitate particles were flocculated and settled out of solution in the 2,000-gallon tank, with some further removal in the clarifying tanks. Midway through the demonstration (October 3), two bag filters were installed in series after the clarifying tanks. These bag filters were used to better prevent fine precipitate particles from flowing back to the plant through the leachant recycle. In spite of these modifications, recycled leachant quality deteriorated and there was a buildup of lead in the system. This was an important factor contributing to the inability of the plant to maintain its performance.

5.2 HYDROCHLORIC ACID PROCESS

Vendor 2 utilized a physical separation module coupled with a leaching module that employed hydrochloric acid as the leachant. The fully assembled processing plant is shown in Figure 5-6. The plant was modular in design and this allowed Vendor 2 to quickly make on-site modifications to the plant setup. The main process units of the plant consisted of a feed hopper and conveyor, a screen deck, an attrition scrubber, two jigs, three sand screws, two clarifying tanks, a clean water storage tank, an organic screen, a precipitation tank, three chemical storage tanks, a centrifuge, two flocculation tanks, and a plate-and-frame filter press.

Figure 5-7 is a process schematic showing the configuration of the various components of the hydrochloric acid process. The plant was installed, piped, and all system connections were made in 2½ weeks, beginning October 21, 1996. On November 8, the plant was filled with 22,000 gallons of water and a leak check was performed to ensure that the acid wash solution would not leak onto the pad. Minor leaks were discovered and corrected for the plant validation test. Prior to the validation test, some raw berm soil was run through the plant to determine if any process modifications were necessary. During these trial runs, it was determined that the screen deck and coarse material jig would be taken off line, and that some minor adjustments to other process units needed to be made before the validation test. No major modifications were made to the plant once mobilization was completed and the validation test was run.
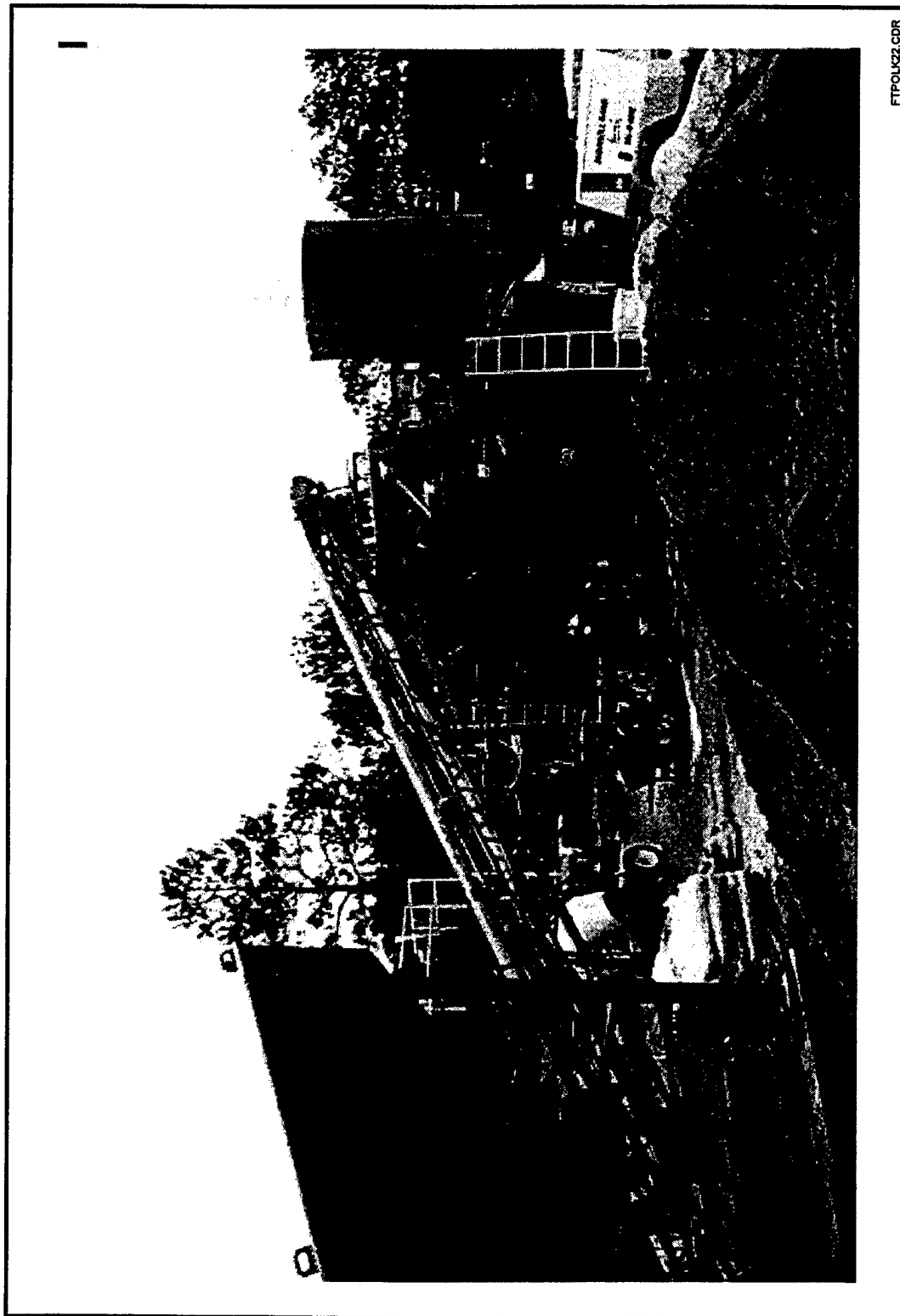
Figure 5-6. Photographic Image of the Hydrochloric Acid Process

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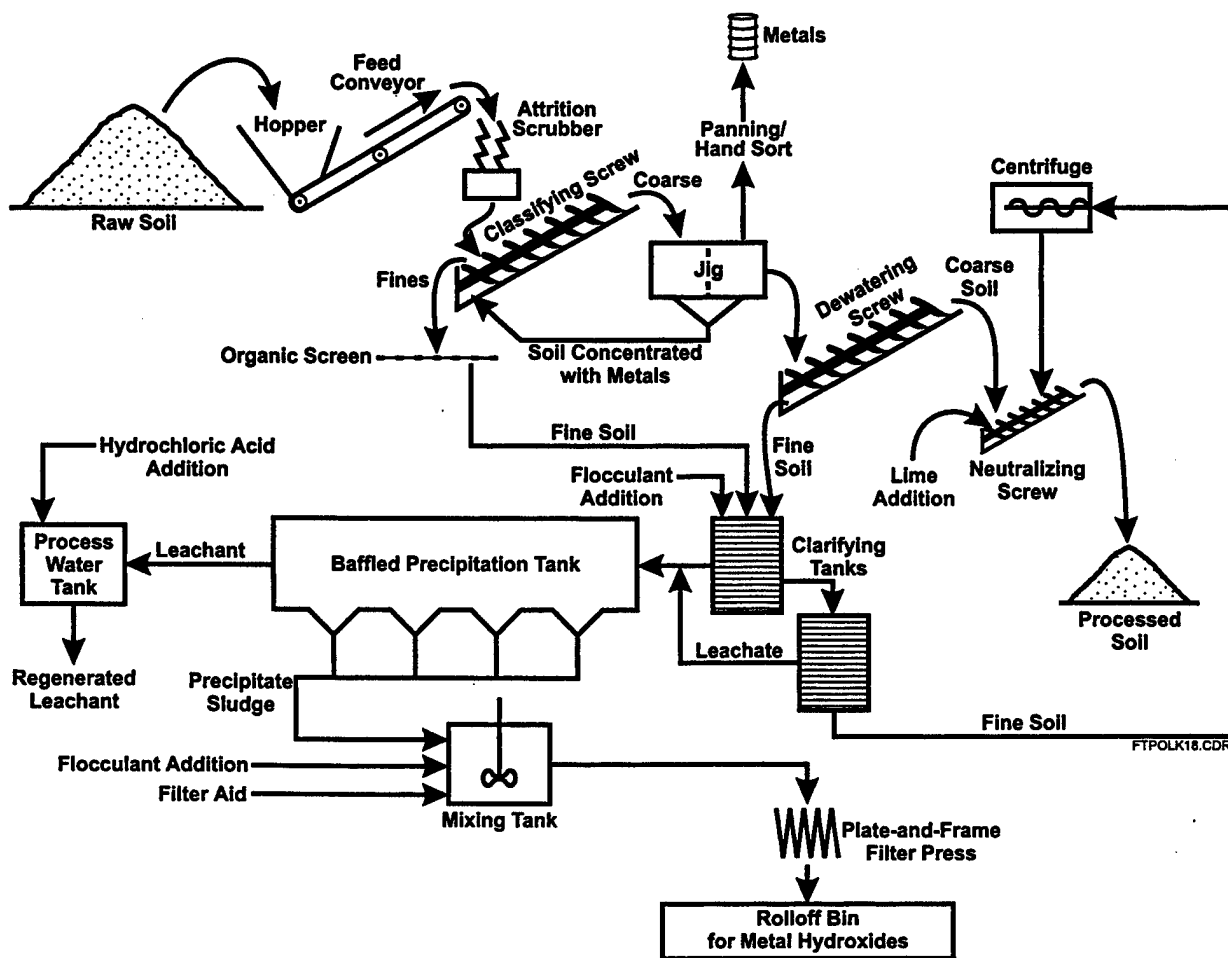
Figure 5-7. Final Schematic of the Hydrochloric Acid Process

5.2.1 Physical Separation Module

The physical separation module is shown in Figure 5-8. Streams that were sampled for this evaluation are also noted in this figure as alphabetical symbols. The physical separation module initially separated the soil into coarse (+200 mesh) and fine (−200 mesh) fractions. The coarse fraction was processed by density separation. The fines were sent to the leaching module.

5.2.1.1 Size Separation

Raw soil from the stockpile was loaded into the feed hopper with a front-end loader and fed to the attrition scrubber via a belt conveyor. The belt conveyor was approximately 34 feet long and was equipped with load cells for determining/ recording feed rates and totals. The hopper and belt feeder worked extremely well with Fort Polk soil. After the soil was slurried with acidic wash solution it was fed to the first spiral classifier (sand screw). The coarse fraction was carried up the sand screw by mechanical rotation, and was discharged to the jig. The overflow, which
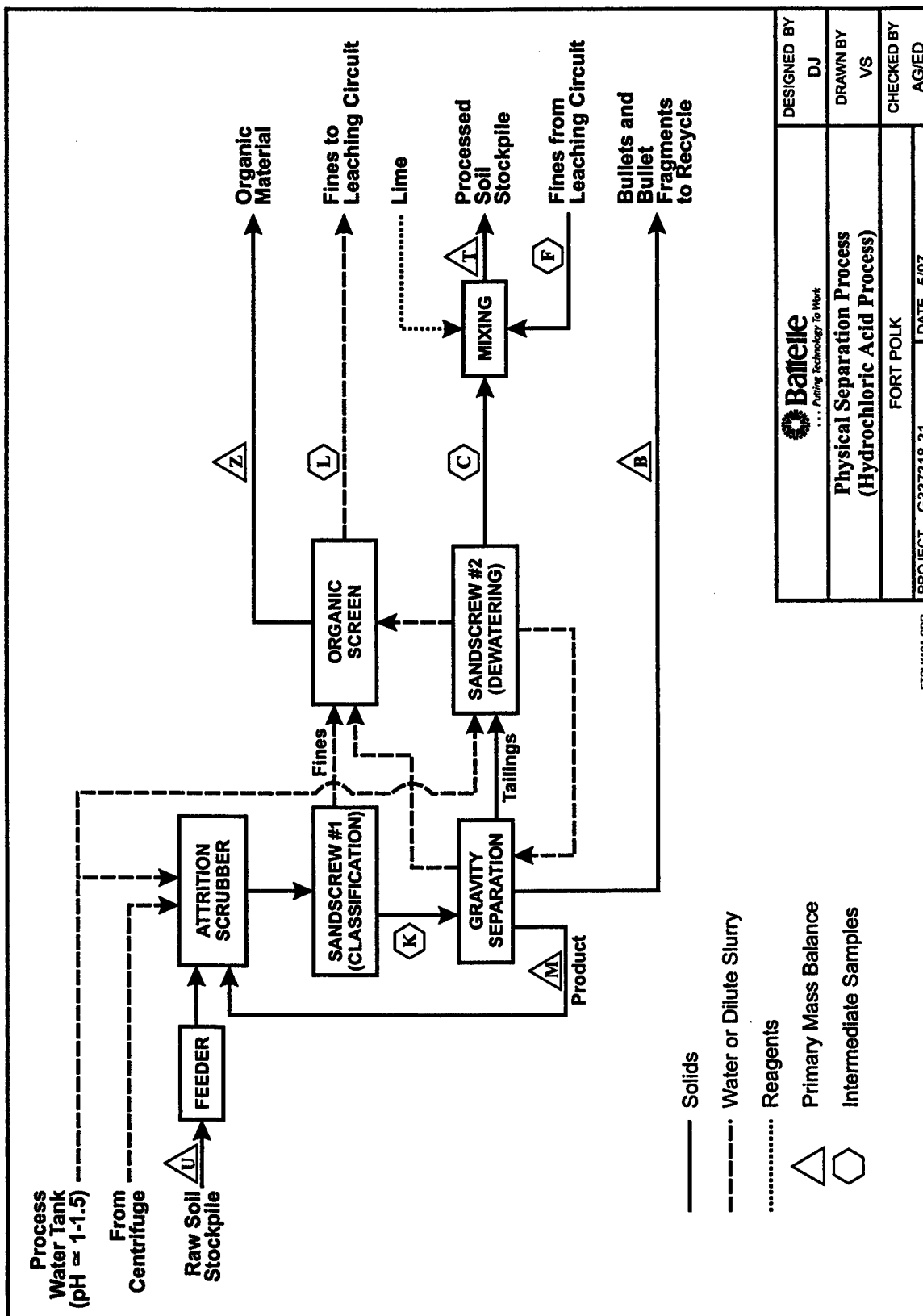
Figure 5-8. Physical Separation Process (Hydrochloric Acid Process)

contained mostly fine soil particles, was sent to a screen to remove organic matter. The under-flow from the screen was sent to the leaching module.

5.2.1.2 Density Separation

After exiting the top of the sand screw, the coarse fraction was fed to the jig. During initial processing, the metal concentrate stream (M) from the jig did not show any significant metals content. Consequently, stream (M) was routed back to the inlet of the sand screw for the rest of the demonstration. This lack of metals in the jig concentrate could be because (1) the raw berm soil did not contain lead in the size fraction amenable to jig separation, or (2) an optimum jig was not used. Several coarse metal fragments accumulated on top of the 1/8-inch slotted punch plate in the jig bed. These fragments were removed and hand-sorted by an operator who was stationed there throughout the demonstration. The raw soil contained some asphalt and concrete gravel, which also accumulated on top of the jig bed and had to be removed manually by an operator. At the end of each day, the metal particulates that had accumulated on the slotted plate were removed and stored in 55-gallon drums for off-site recycling. A punch plate with larger slots probably would have allowed the jig to operate more efficiently.

The overflow from the jig bed was transferred to the second sand screw. This screw provided additional acid contact time for the coarse fraction, dewatered the coarse fraction, and removed additional fines from the coarse fraction and sent them to the leaching circuit. The pitch of this sand screw was raised to approximately 35° to aid in the dewatering of the coarse fraction. The discharge from this sand screw was fed into the mixing/neutralizing sand screw where it was mixed with the leached fines and neutralized with hydrated lime powder to a pH of 5.5. An operator routinely added hydrated lime to this last sand screw to neutralize excess acid in the processed soils.

5.2.2 Leaching Module

Figure 5-9 shows the leaching module configuration. Hydrochloric acid at a pH of 1.5 was used as both the wash solution in the physical separation circuit and as the leachant in the leaching module. The leaching module consisted of two clarifying tanks, a baffled precipitation tank, two mixing tanks, and two water storage tanks. The following subsections describe the acid leaching and precipitation process used by Vendor 2.

5.2.2.1 Acid Leaching of Fine Soils

The slurry containing the fines (−200 mesh) from the physical separation module was fed to the first clarifying tank. In this tank, flocculant was added and the fines were given time to leach. The overflow from this clarifier was pumped to the precipitation tank. The underflow from the first clarifier was fed to the second clarifying tank where it was mixed with fresh hydrochloric acid. Each tank was equipped with 45° angled plates. This allowed the fine soil particles to roll down to the bottom of the tanks from where the resulting sludge was pumped to the next tank. The sludge from the second leach tank was sent to a bowl centrifuge, where it was dewatered. The dewatered solids from the centrifuge were sent to the mixing/neutralizing screw. In this
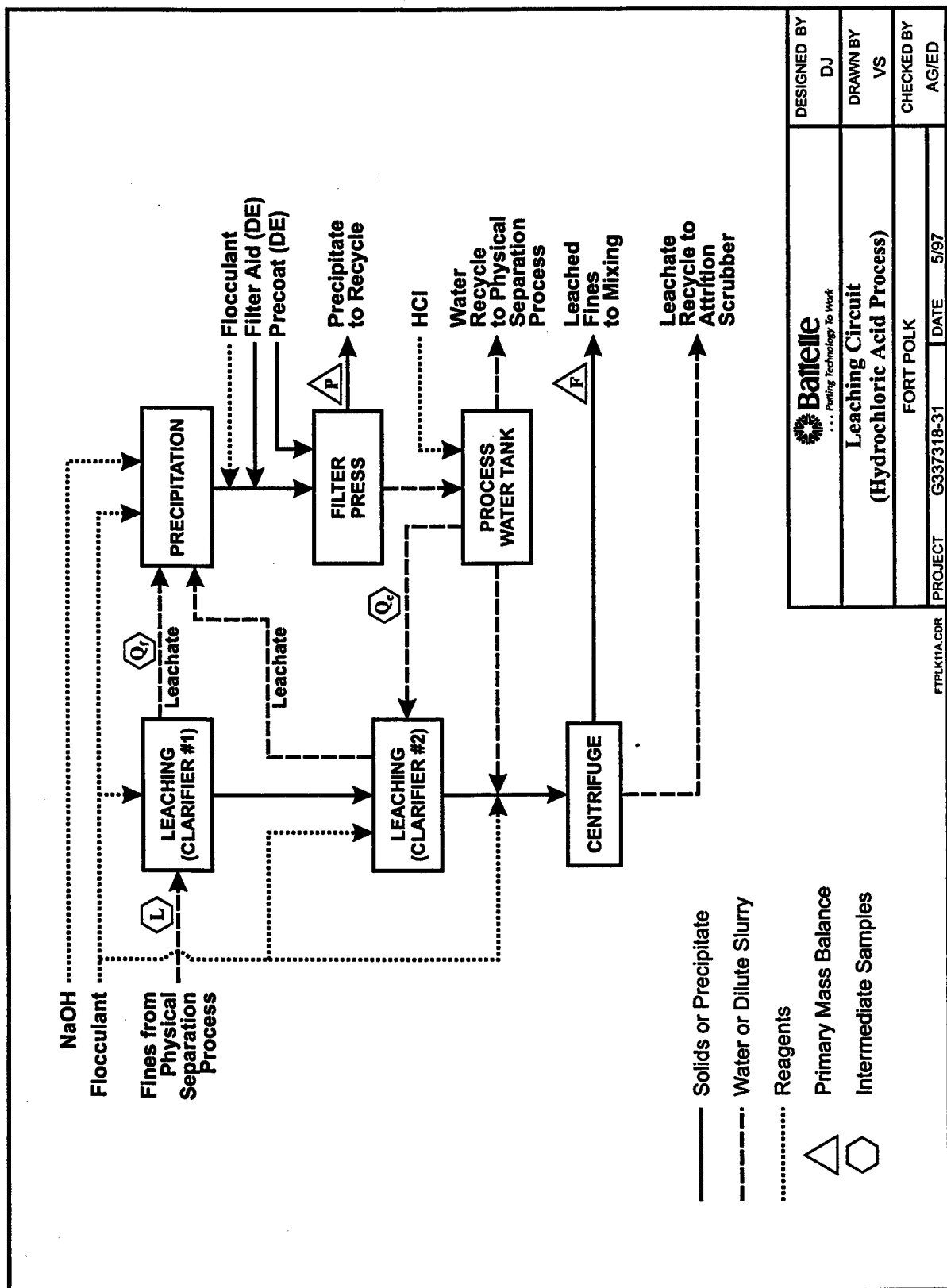
Figure 5-9. Leaching Circuit (Hydrochloric Acid Process)

screw, the fines were recombined with the coarse processed soil fraction, and the pH was raised to the native soil level of about 5.5. The spent leachant containing soluble metals was pumped from the second clarifying tank to the precipitation tank.

5.2.2.2 Precipitation and Regeneration of Leachant

The precipitation portion of the plant is also shown in Figure 5-9. The precipitation portion consisted of a large four-tiered compartment with baffles, two mixing tanks in series, and a plate-and-frame press. Sodium hydroxide was used as the precipitant and was introduced into the baffled precipitation tank. Sodium hydroxide and a flocculant were added to the tank to raise the pH to 9.5. Metals were precipitated out at the higher pH. The metal precipitate particles formed flocculates until they were large enough to sink to the bottom of the tank. From there, the precipitate sludge was pumped to the mixing tanks where it was stored and mixed until the end of the day. At the end of each day, the sludge was pumped to a mixing tank and mixed with diatomaceous earth to improve filterability. From the mixing tank, the sludge was pumped to the plate-and-frame filter press, which was also precoated with diatomaceous earth to ensure that the filter cloths would not clog. After the filter press had dewatered the precipitate sludge, the plates were scraped clean into a roll-off bin by operators. Three roll-off bins were filled during the demonstration. These bins were sent to a smelter for off-site recycling.

The spent leachant from the precipitation tank overflowed to the process water tank, where it was reacidified with fresh hydrochloric acid to a pH of 1.5. The spent leachant liquid from the filter press described above was stored in a 3,000-gallon cone-bottom water tank. From there, it was pumped to the process water tank for regeneration. From the process water tank, samples were taken periodically by the vendor to ensure that the lead levels in the regenerated leachant were low.

6. Evaluation Methodology Used for the Demonstration

The evaluation of the two vendors' processing was conducted by Battelle through field observation, collection and preparation of appropriate samples on site, and off-site laboratory analyses.

6.1 OBJECTIVES OF SAMPLING AND ANALYSIS

Because of the time and resources associated with collecting and processing the large sample sizes required, the field sampling team was increased to three personnel. The objectives of the sampling and analysis effort were prioritized as follows to better meet demonstration goals with the available resources:

☐ The primary objective of the sampling was to ensure that the two critical process performance objectives (namely, TCLP and total lead in the final processed material) were met. This critical sampling objective had to be met daily or for every 80-ton batch of raw soil stockpile to determine whether or not the final processed soil was suitable for return to the berm.

☐ A secondary objective was to evaluate the lead removal efficiencies of the two major elements of the process (physical separation and acid leaching).

☐ Other objectives included evaluating the lead removal efficiencies of individual process units (e.g., jigs), and evaluating the removal of other undesirable metals (antimony, copper, and zinc) in the various process streams.

6.2 PROCESS STREAMS SAMPLED

The two vendors' plants may be described by the simplified schematic shown in Figure 6-1. All the input and output streams to the two major process elements, separation and leaching, are indicated. Sampling locations for the two vendors' processes are shown in Figures 5-4, 5-5, 5-8, and 5-9 (in Section 5). The process streams sampled are labeled using capital letters of the alphabet. The final processed soil that was returned to the berm is labeled 'T.' The sampling and monitoring schemes listed below were followed once steady state (or at least a reasonably continuous period of processing) was achieved:

☐ Raw and Final Processed Soil: Each batch of processed soil (T) was sampled and analyzed (TCLP and total metals) to ensure that target performance criteria were met prior to returning the material to the berm. Each batch of raw soil (U) delivered to the processing area was also sampled and analyzed to determine the concentrations and variability of metal contaminants in the feed to the plant.

☐ Input and Output Streams: Where possible, the input and output streams to the physical separation (C, M) and leaching (L, F, and P) circuits were sampled and analyzed to evaluate the removal efficiency of these two major elements of the process. For Vendor 2, whose plant reached steady state, a rough mass balance was attempted with these data.

PROCESSED
SOIL
T (Blend)

COARSE SOLIDS
FROM
PHYSICAL
SEPARATION **C**

FINE SOLIDS
FROM
.LEACHING **F**

TREATED
MATERIAL

RAW
SOIL **U**

FEED

PHYSICAL
SEPARATION
CIRCUIT

L

LEACHING
CIRCUIT

FTPOLK02A.CDR

METALS

O **M**

P

OVERSIZE
MATERIAL

UNDERFLOW
FROM
JIG

PRECIPITATE
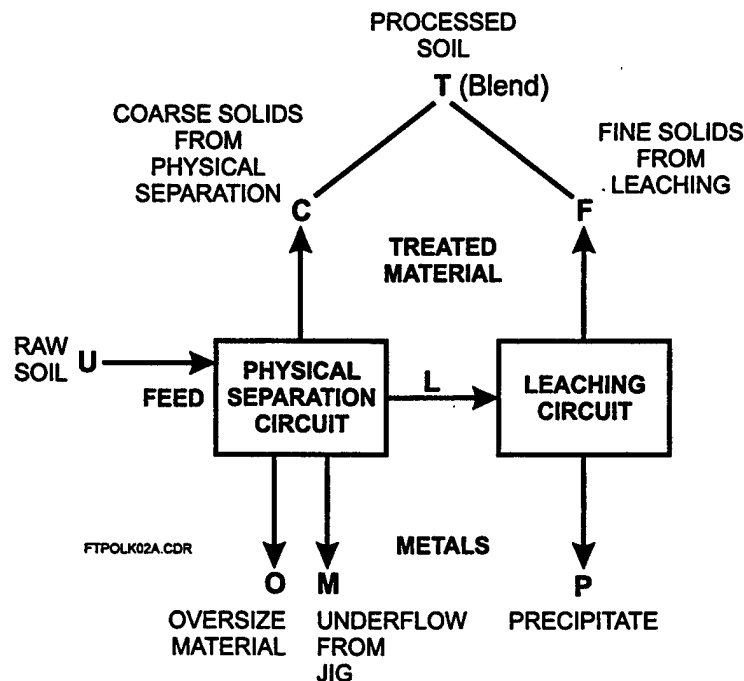
Figure 6-1. Schematic of Acetic Acid Process Showing Input and Output Streams

☐ Intermediate Streams: Where possible, the input and output streams to selected individual process units (e.g., jigs) were sampled and analyzed for total metals to evaluate the removal efficiency of individual units.

6.3 ENSURING THE REPRESENTATIVENESS OF SAMPLES

Obtaining representative samples from heterogeneous process streams was the main sampling challenge. When particulate metal contaminants are present, the "nugget" effect makes sampling difficult. A single metal fragment, depending on whether or not it is captured in the sample bottle, can dramatically alter the analytical result. The larger the possible metal fragment size, the larger the sample volume required to minimize this effect.

To obtain representative samples, the sampling methodology shown in Figure 6-2 was used. For a given process stream, once the process reached steady state, grab samples were taken and combined to form a composite sample that was large enough to be representative of the maximum particle size present. The sample size of the composite was based on Table 6-1, which is a statistical reference for sampling in the mining industry (Taggart, 1945).

For example, based on Table 6-1, between 42 and 1,800 lb of material would be required if the largest metal particle was expected to be ⅜ inch (0.375 inch), depending on the grade of ore (distribution of metal in the matrix). For a medium ore, about 300 lb (approximately 30 gal) of material would be needed to account for the maximum possible particle diameter of ⅜ inch (0.375 inch in the table) that could be expected. Statistically, this would afford over 99%
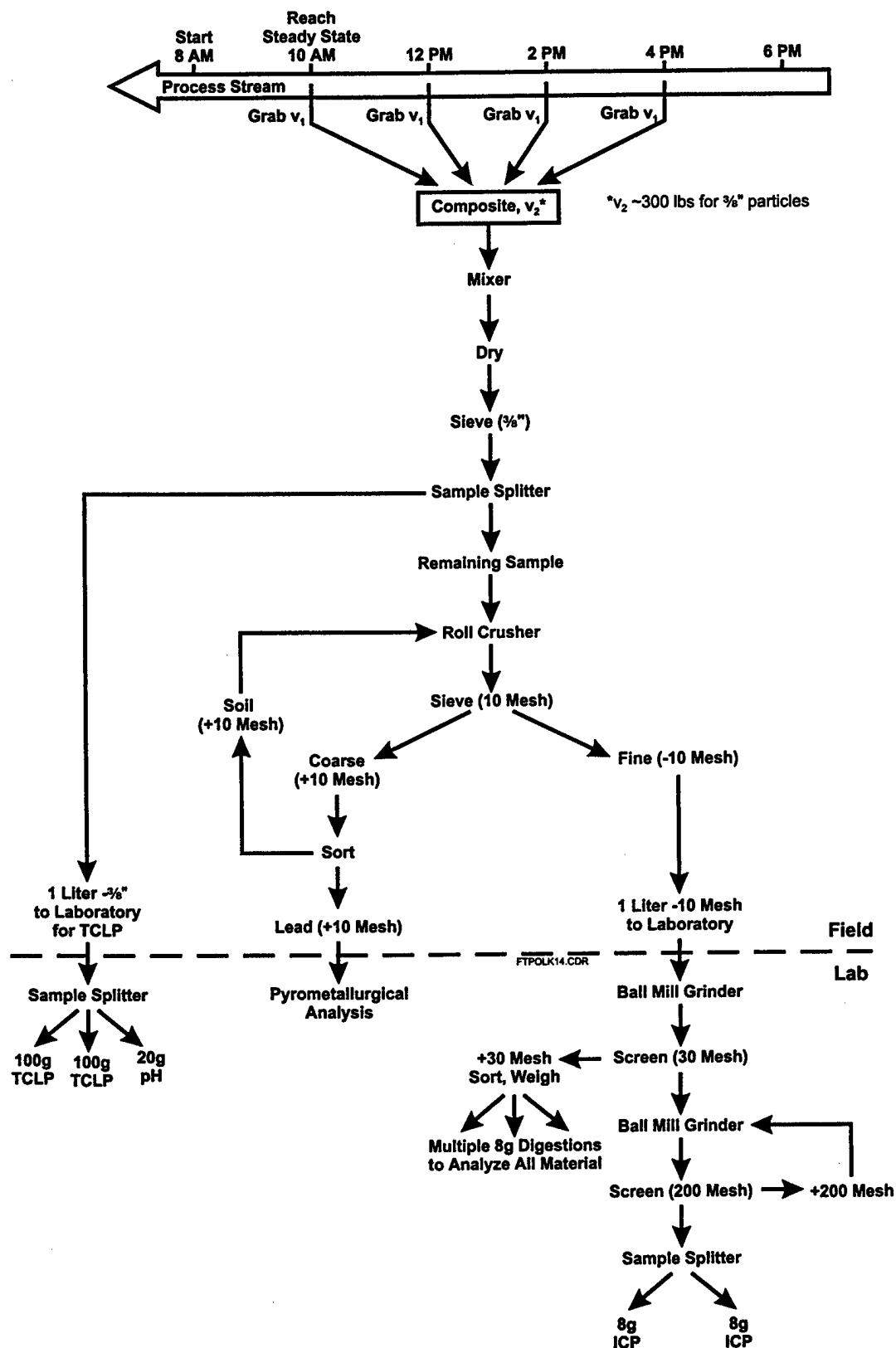
Figure 6-2. Sampling Methodology for Each Primary Process System

**Table 6-1. Mass (in Pounds) of Composite Required to Obtain
Representative Samples**

Diameter of Largest Piece			Soil Grade				
Inches	mm	Mesh	Very-low-grade or very uniform ores	Low-grade or uniform ores	Medium ores		Rich or spotty ores
8	-	-	19,200	64,00	-	-	-
6	-	-	10,800	36,000	80,000	-	-
5	-	-	7,500	25,000	55,550	-	-
4	-	-	4,800	16,000	35,556	80,000	-
33	-	-	2,700	9,000	20,000	45,000	-
2.5	-	-	1,875	6,250	13,888	31,250	80,000
2	-	-	1,200	4,000	8,889	20,000	51,200
1.5	-	-	675	2,250	5,000	44,580	28,800
1.25	-	-	496	1,536	3,472	7,813	20,000
1	-	-	300	1,000	2,222	5,000	12,800
0.75	-	-	169	536	1,250	2,813	7,200
0.625	-	-	117	391	868	1,953	5,000
0.500	-	-	75	250	556	1,250	3,200
0.375	-	-	42	141	313	704	1,800
0.3125	-	-	29	98	217	488	1,250
0.250	-	-	19	63	139	313	800
0.1875	-	-	10.5	35	78	176	450
0.131	3.327	6	5.15	17.2	38.1	86	220
0.093	2.362	8	2.6	8.65	19.2	43	111
0.065	1.651	10	1.29	4.3	9.5	21.5	55
0.046	1.168	14	0.65	2.16	4.8	10.75	28
0.0328	0.833	20	0.322	1.075	2.37	5.38	13.76
0.0232	0.589	28	0.162	0.539	1.20	2.69	6.90
0.0165	0.417	35	0.081	0.269	0.59	1.345	3.44
0.0116	0.295	48	0.041	0.135	0.30	0.673	1.73
0.0082	0.208	65	0.020	0.067	0.15	0.336	0.86
0.0058	0.147	100	0.010	0.034	0.075	0.168	0.43
0.0041	0.104	150	0.005	0.017	0.038	0.084	0.215
0.0029	0.074	200	0.0025	0.009	0.019	0.042	0.107

Adapted from Taggart, 1945.

confidence that the lead concentration in the composite sample represents the true mean of the population (original process stream). This holds if the entire 300-lb sample can be extracted and analyzed. This would be beyond the scope of generally available analytical techniques.
However, according to EPA Standard Methods 3051 and 1311, total metals analysis starts with a 2-g aliquot and TCLP analysis starts with a 100-g aliquot. The challenge was to obtain small representative aliquots of material from the 300-lb (30-gal) composite to get an acceptable level of confidence (80% targeted) in the result.

As indicated in Table 6-1, the required sample size decreases as particle size decreases. After trying out several sample preparation schemes, it was decided that size reduction would be the best way to obtain successively smaller representative aliquots. After modifications to the

microwave digestion procedure, the largest possible sample for a single acid digestion was determined to be 8 g. The largest particle size that allows an 8-g sample to be representative of a medium ore is 200 mesh. Therefore, an approach was needed to process a large composite sample to decrease particle size. Using repeated cycles of size reduction and sample splitting to reduce sample size while controlling the fundamental sampling error within specified limits is a well-developed practice in the mineral processing industry (Gy, 1982; Pitard, 1992). Size reduction of small-arms range soils is complicated by the malleability of lead which causes lead metal to deform and smear rather than break apart when subjected to size reduction processing. The sample preparation approach devised for the Fort Polk demonstration, illustrated in Figure 6-2, attempted to isolate and weigh such flattened pieces of lead collected during grinding.

6.3.1 Collecting the Composite

The first phase of the sampling process was to collect a representative composite sample weighing about 300 lb. The method used to collect the composite ideally would include statistical design with equal probability of sampling any point in the total volume. The ideal case is collecting samples at random points in a one-dimensional stream. Good practical implementations of this ideal are collecting samples at random points throughout a waste pile or berm as the material is moved or by cross-stream sampling of a moving stream, e.g., soil on a conveyor belt. Good composite samples of a static pile or berm are difficult to collect because it is hard to provide an equal probability of reaching any part of the volume (Pitard, 1992). Composite samples from a stockpile or berm can be collected using core samples taken at random locations, but the sampler must be careful to collect core through the entire pile depth.

For the Fort Polk demonstration, the raw soil composite was collected by taking several grab samples from various points in the pile of berm soil delivered to the pad for processing. Because the pile was too large to collect cores from, periodic groups of grabs were collected as layer after layer of the pile was uncovered by the front-end loader that transferred the soil to the feed hopper. Because of the slow plant processing speeds, especially for Vendor 1, the raw soil composite (300 lb) had to be collected over a period of 2 or more days. The processed soil (T) pile was sampled similarly to collect the 300-lb composite.

Other process streams, such as C, where larger metal particulates could be present were also sampled through large composites. For Vendor 1, the composite size was 300 lb. Because of low variability in the replicates analyzed by this method, and because Vendor 2 was planning to initially screen the soil with a smaller (¼-inch) screen, the composite size was reduce to 150 lb. Process streams with smaller expected particle sizes, such as L, F, and P, were sampled through much smaller composites.

6.3.2 Field Sample Preparation to Reduce the Composite

The composite sample was mixed in a cement mixer, dried overnight in a large (7-ft-tall) on-site oven, and passed through a ⅜-inch sieve. Essentially all of the material passed through the sieve, so no oversize processing was required. The well-mixed, sieved composite was then subsampled to collect material for the TCLP test. Two or three 1-L aliquots of the material

passing through the ⅜-inch screen were collected in bottles and shipped overnight to the off-site laboratory for TCLP and pH analysis. Size reduction smaller than ⅜ inch is not recommended by the TCLP standard method. A riffle-type sample splitter was used to split the composite into equal 1-L aliquots. When the sample bottles were received by the off-site laboratory, a riffle splitter was again used to obtain duplicate 100-g aliquots used for the TCLP procedure.

The remainder of the 300-lb composite was processed on site in a roll crusher to reduce the particle size so that representative 1-L (about 4-lb) samples could be collected and sent to the off-site laboratory. Material remaining on a 10-mesh sieve after crushing was hand-sorted to identify flattened bullet metals that would not break up in the crusher. The bullet metals were sent to a mining laboratory for analysis by pyrometallurgical methods. As seen in Table 6-1, for a uniform material ground to − 10 mesh, the representative sample size is approximately 4 lb (1 L).

6.3.3 Off-Site Laboratory Sample Preparation to Reduce 1-L Samples

Additional size reduction of each 1-L sample was done by the laboratory in a ball mill. The sample was ground to −200 mesh, at which size a 4- to 8-g sample becomes representative. Some material was very difficult to grind smaller than 30 mesh. Consequently, the small +30 mesh subsample was isolated. This +30-mesh material was digested in 8-g batches and analyzed by inductively coupled plasma (ICP) for total metal content. Two 8-g splits were collected from the ground subsample (−200 mesh), digested, and analyzed for total metal content by ICP.

The final total metal concentration result for the initial 300-lb composite sample was determined as the weighted average of the metals concentrations in the +10 mesh subsample (oversize in the field grinding), in the +30 mesh subsample (oversize in the laboratory grinding), and the −200 mesh subsample.

6.4 ANALYTICAL METHODS

Standard EPA Method 1311 was followed for TCLP analysis of soil samples. Standard EPA SW-846 Method 3051 was generally used for digestion of samples for total metals analysis, with a few modifications. The sample size for digestion was increased from 2 g to 8 g to enhance the representativeness of samples that may contain particulate metals, a situation not considered in the EPA Standard Method. The 8-g sample size was the maximum that could be accommodated in the sample cups of the microwave digester. The acceptability of the larger sample size was verified in a preliminary method development effort. Another modification was made to improve the recovery of antimony by using hydrochloric acid, as well as nitric acid, for the digestion. The digestates were analyzed by ICP according to EPA SW-846 Standard Method 6010.

Special pyrometallurgical analysis was conducted on the predominantly metal fragments (+10 mesh) isolated during sample preparation in the field. This type of analysis uses a reduction

and slagging approach, similar to full-scale smelting, to separate metals from the silicate soil matrix. Weighted amounts of fluxing agents such as borax and soda ash and a carbon source to promote reduction are mixed with the sample. The sample is heated to >538°C (1,000°F) to form a molten slag layer that separates and floats on a molten metal layer. After cooling, the slag and metal are separated and the two fractions are weighed. Metal shavings are collected, dissolved, and analyzed and the slag is pulverized, digested, and analyzed to determine the metal content of the two fractions. The total metal content of the +10 mesh sample can be determined using the weight and composition of the two portions. The total metal contents of the +10 mesh fractions isolated from raw soil (U) samples and the recovered metals stream (O) were determined by this method.

6.5 ALTERNATIVE SAMPLING AND ANALYSIS SCHEME

A preliminary comparison was made of the composite sampling methodology described above and an alternative method that consisted of taking small grab (core) samples from the large composite and analyzing each grab separately by the sample preparation portion shown in Figure 6-2 for the 1-L samples. This preliminary comparison was done for the processed soil (T) sample of September 15, 1996, as shown in Table 6-2. The large composite TCLP result was 3.07 mg/L, and the grab samples averaged 3.08 mg/L. The average total lead result was 122 ppm for the large composite sample and averaged 119 ppm for the grab samples. The final total lead results were in agreement; the difference was that the relative standard deviation (RSD) for the grab samples was slightly higher. Taking multiple grab samples of sufficient size (4 lb or more) and analyzing each separately is a more cost-effective method that may be useful at future sites for evaluating routine range maintenance activities.

Table 6-2. Comparison of the Analytical Data of Large and Grab Samples

Sample ID	Sampling Method	Total Lead Result (mg/kg)	TCLP Lead Result (mg/L)
September 15, A	Large Composite	N/A	3.11
September 15, B	Large Composite	N/A	2.98
September 15, C	Large Composite	N/A	3.10
September 15, D	Large Composite	123.3	N/A
September 15, E	Large Composite	120.3	N/A
Average	Large Composite	121.8	3.06
Percent RSD	Large Composite	1.74%	2.36 %
September 15, X	Small Grab	114.2	3.18
September 15, Y	Small Grab	116.9	3.04
September 15, Z	Small Grab	125.6	3.01
Average	Small Grab	118.9	3.08
Percent RSD	Small Grab	2.95%	5.01 %

N/A = Not applicable/available.

6.6 ON-SITE XRF ANALYSIS

In order to quickly check each vendor's process performance, on-site testing using an XRF analyzer was performed on some samples. During pre-demonstration meetings with the Army and the Navy, arrangements for the on-site testing capability were made. An XRF analyzer was rented from TN Technologies, Inc. The unit, a Spectrace™ 9000 XRF, was used for on-site analyses of lead, copper, zinc, and antimony in soil samples. Soil samples were prepared for the XRF by two distinct methods, depending on how soon the results were required, as described below:

❑ Wet Method: For faster turnaround, samples were collected from the process streams with a stainless steel spoon and analyzed wet. Approximately 10-g aliquots of the sample were placed in Teflon™ cups, which were then sealed with a Mylar™ film. The filled cups were inverted (film side facing the analyzer) and placed on the XRF analyzer, and the analysis was performed.

❑ Dry Method: Samples were collected from the sampling streams with a stainless steel spoon. The samples were then dried in an oven at approximately 105°C. After drying, the samples were ground to -10 mesh. Approximately 10-g aliquots of the sample were then put in Teflon™ cups and sealed with a Mylar™ film. Samples were placed on the XRF analyzer and the analysis was performed.

Upon completion of the XRF analyses, the results of the data were downloaded via RS 232 cable to an on-site portable computer. The XRF data are included in Appendix H (Tables H-1 and H-6). No appreciable difference was attributed to the XRF analyses performed using either of the two preparatory methods, although the dried and ground samples normally gave higher concentration averages.

A comparison of the on-site XRF and off-site laboratory ICP analysis of processed soil (T) samples is shown in Figure 6-3. Tables H-2 through H-5 and H-7 through H-10 in Appendix H detail the XRF and ICP analysis averages and standard deviations for various process streams sampled during both demonstrations. As can be seen from these tables, the sample preparation and analysis by ICP is more accurate for total metals analysis, especially in process streams that are nonhomogeneous. Based on this comparison, the XRF unit appears to be useful as a field screening tool for providing an indication of lead levels present in matrices that are fairly homogeneous, such as the processed soils (T), soil fines (F), and the precipitate (P). Even though the lead concentrations were high in the precipitate, the XRF data indicated the high lead levels as did the ICP data. For samples that had more particulate metals, such as the raw soil (U) and jig feed (K), the XRF analyses did not give a good indication of lead content. Because metal particulates tend to shield one another and the 10-g aliquots used for XRF analysis are not representative, there is the potential for underestimating the lead content of the sample by XRF analysis. In general, as demonstrated by Vendor 2, an on-site AA unit is probably more useful and desirable for process verification during small-arms range soil processing.

(a) Acetic Acid Demonstration
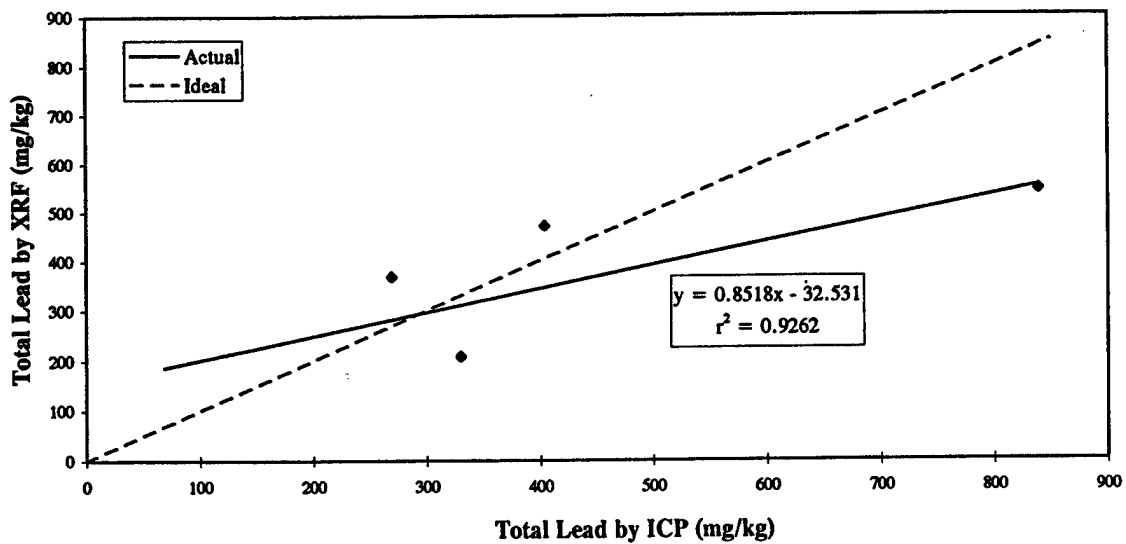
(b) Hydrochloric Acid Demonstration
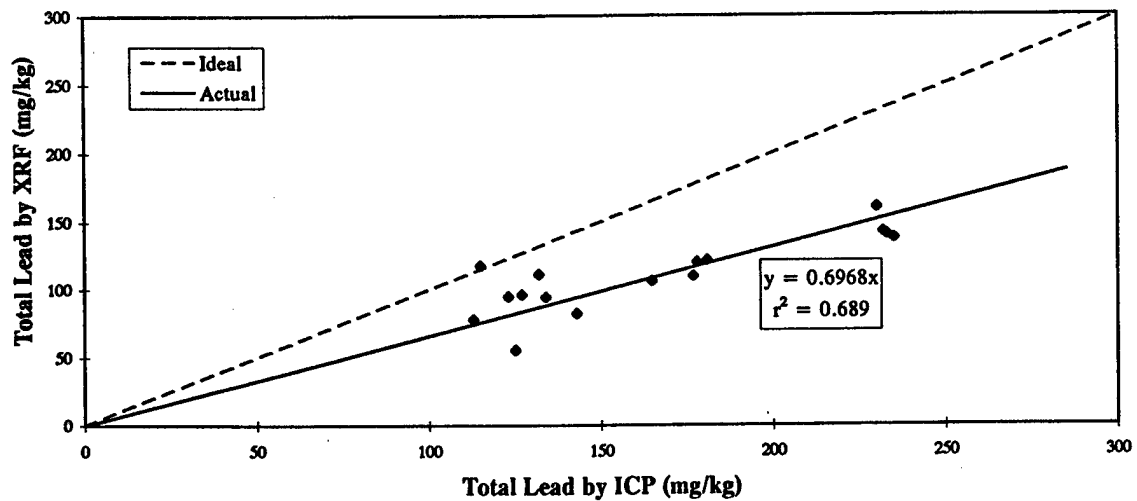
Figure 6-3. Comparison of XRF and ICP Analyses of Lead in the Processed Soil Using (a) Acetic Acid and (b) Hydrochloric Acid

7. Demonstration Results

The performance of the two process plants was evaluated in terms of both technical and cost criteria. The technical performance of the two plants was evaluated by examining the following issues during the demonstration:

☐ Ability of each plant to remove total and leachable lead to target levels in the processed soil. The target for total lead in the processed soil was 1,000 mg/kg for Vendor 1 using acetic acid and 500 mg/kg for Vendor 2 using hydrochloric acid. The target was reduced for Vendor 2 to better meet the leachable lead criterion. The target for leachable lead in the processed soil was 5 mg/L (TCLP) for both vendors.

☐ Types (hazardous or nonhazardous) and quantities of residuals generated by the two plants. Acceptability of metals concentrate streams for off-site recycling.

☐ Ability of each plant to reach and maintain steady state at conditions determined by the bench-scale treatability testing and scale-up design.

☐ Ability of each plant to remove other undesirable metal contaminants, such as copper, zinc, and antimony. Although lead is the main regulatory concern at small-arms range sites, the removal of other heavy metals is also desirable. In some states, such as California, copper, zinc, and antimony are environmentally regulated metals.

☐ Physical and chemical suitability of the processed soil for reuse in the berm.

The cost-effectiveness of the two plants was evaluated by estimating fixed (capital) and variable (operating) costs incurred during the demonstration.

7.1 PERFORMANCE OF VENDOR 1 AND THE ACETIC ACID PLANT

The process streams that were sampled to evaluate Vendor 1's plant performance are described in Figures 5-4 and 5-5 (Section 5.1). Appendix F contains the detailed analytical data for the samples collected during Vendor 1's demonstration.

7.1.1 Lead Removal by Vendor 1 Process

Table 7-1 shows that during the plant validation test on September 15, 1996, which was the first day of processing, the total lead content of the range soil was reduced from 1,854 to 122 mg/kg, a 93% removal of lead. On this first day, the leachable lead content of the soil was reduced to 3 mg/L, which is below the TCLP target of 5 mg/L. This would seem to indicate that the use of acetic acid has the potential to reduce lead in some small-arms range soils to below target levels. On subsequent days, however, the level of lead in the processed soil continued to rise incrementally. The process efficiency of the plant declined as the demonstration progressed. On October 4, the processed soil collected on September 21 and 25 was reprocessed through the plant in an attempt

**Table 7-1. Overall Removal of Total and Leachable Lead with the
Acetic Acid Process**

| | Total Lead | | | TCLP Lead | |
Date	Raw Soil (mg/kg)	Processed Soil (mg/kg)	Removal (%)	Raw Soil (mg/L)	Processed Soil (mg/L)
15-Sep	1,854	122	93	34.6	3.07
21-Sep	1,407	208	85	21.0	5.99
25-Sep	3,347	330	90	22.0	10.3
2-Oct	2,741	404	85	40.5	11.2
4-Oct[a]	208 - 330	269[b]	None	5.99 - 10.3	7.80[b]
10-Oct	4,789	839	82	106	21.7
12-Oct	4,789	1,443	70	106	48.0
Statistics					
n	5	N/A	N/A	5	N/A
Avg.	2,828	N/A	N/A	45	N/A
Std. Dev.	1,331	N/A	N/A	35	N/A
80% C.I.	2,828±792	N/A	N/A	45±21	N/A

(a) This sample is a combination of the processed samples from September 21 and 25 that failed TCLP testing.
(b) These sampling data are the results of reprocessing of soil that failed TCLP on September 21 and 25.
N/A = Not applicable. The process did not reach steady state and the distribution is not normal.
C..I. = confidence interval.
n = Number of independent measurements.

to meet the TCLP criterion. Table 7-1 shows that reprocessing did not help. The total and TCLP lead concentrations in the reprocessed soil are almost exact averages of the concentrations in the processed soil piles collected on September 21 and 25.

Table 7-2 shows the stream assays and pH ranges of the various process streams over the course of the acetic acid demonstration. The process streams U, T, M, O, P, and Z represent the input and output streams of the process plant. This table shows that most of the coarse particulate metals that were removed from the soil were collected in the blade mill (as part of Stream O) rather than in the screen deck or jig (M). The coarse fragments that settled in the blade mill had an assay of almost 24% lead. The jig concentrate (M) did not contain much metal. As described in Section 3.2.1, the finer particulate lead fraction in the soil may not have been amenable to gravity separation. The precipitate sludge (P), which had a lead assay of 1.2%, contained the metals removed from the spent leachant during precipitation treatment. The organic matter (Z) that was screened from the blade mill overflow had a high concentration of lead, but because this stream was relatively small the total lead mass recovered in Z was not very high.

As can be seen in Table 7-2, the TCLP lead in either the coarse (C) or fine (F) processed soil stream during the October 1-3 sampling period was above 5 mg/L. This indicates that both fractions contributed to the elevated TCLP result. The leaching module did manage to reduce the total lead in the fines from 5,347 mg/kg (L) to 947 mg/kg (F) and the leachable lead content from 49.9 mg/L (L) to 15.1 mg/L (F).

Table 7-2. Distribution of Lead in Acetic Acid Process

Process Stream	Stream Description	pH Range	Total Lead Result (mg/kg)			TCLP Lead Result (mg/L)			Avg. Total Lead Conc. (mg/kg)
			Sep. 15	Oct. 1-3	Oct. 7-11	Sep. 15	Oct. 1-3	Oct. 7-11	
U	raw soil	4.0-4.8	1,854	2,741	4,789	34.6	40.5	106	2,828
T	processed soil	4.1-4.9	122	404	839	3.07	11.2	21.7	722
M	jig concentrate	4.8	N/A	484	N/A	N/A	17.6	N/A	484
O	oversize fraction	5.2	N/A	N/A	239,000	N/A	N/A	N/A	239,000
P	precipitate sludge	3.2-3.6	N/A	N/A	11,990	N/A	N/A	321	11,990
Z	organic matter	N/A	6,457	N/A	N/A	11.1	N/A	N/A	6,457
C	coarse processed fraction	4.9-5.5	N/A	252	N/A	N/A	6.49	N/A	252
F	fine processed fraction	4.0-4.3	N/A	947	N/A	N/A	15.1	N/A	947
L	leach circuit feed	4.4	832	5,347	N/A	21.3	49.9	N/A	3,090
Q[a]	regenerated leachant	2.9-3.3	N/A	627	29.3	N/A	N/A	N/A	328

(a) Leachant concentration measured in units of mg/L.
N/A = Not applicable/available.

The regenerated leachant (Q), collected after precipitation treatment, still contained high concentrations of lead, as high as 627 mg/L at one point. This was the main contributing factor to the poor process performance. Inadequate precipitation of lead from the leachant reduced the leaching efficiency of the acetic acid process. For this reason, reprocessing of the processed soil on October 4 did not help. Some efforts were made to improve regenerated leachant quality toward the end of the demonstration. However, on October 7, the regenerated leachant still had 29 mg/L of lead. These lead levels in the regenerated leachant represent dissolved lead concentrations because the leachant samples were filtered before analysis. The pHs of the precipitate sludge (3.2 to 3.6) and the regenerated leachant (2.9 to 3.3) before refortification with fresh acid indicate that the precipitation step was implemented at a very low pH. Although the manufacturer of the proprietary precipitant (ThioRed®) claims that the precipitant works at all pHs, a pH of 8.5 is recommended for optimum precipitation (ETUS, Inc. Marketing Information). One reason the pH was not raised in the precipitation tank was concern about the cost of the large amount of acetic acid that would be required to lower the pH again in the regenerated leachant. The uncertainty about optimum precipitant dose and optimum pH range may have contributed to inadequate precipitation.

In an effort to see if additional dewatering or neutralization of the processed soil to elevated pH would have helped meet TCLP targets, some reserve samples of the processed soil collected on October 10 were subjected to additional adjustments in the laboratory before TCLP analysis. Table 7-3 gives the results of these experiments. In some cases, the processed soil was washed with water to remove excess leachate that might have contained solubilized lead. In other cases, the processed soil samples were neutralized with a lime slurry to incrementally higher pH levels of about 6, 8, and 11. Neither treatment enabled the processed soil to meet the TCLP criterion.

**Table 7-3. TCLP Results of Water Washing and pH Adjustment
on Processed Soil**

Sample ID	pH Adjustment	TCLP Result (mg/L)
C-OC10-T-1A	None	21.7
C-OC10-T-1B	6.0	23.6
C-OC10-T-1C	8.0	15.8
C-OC10-T-1D	11.0	14.9
C-OC10-T-WW	Water washed	17.8

7.1.2 Vendor 1 Process Residuals

Residuals generated during processing and their costs for disposal/recycling are shown in Table 7-4 and include the following:

☐ Recovered Metals: Metal particulates recovered from the screen oversize fraction and from the basin of the blade mill were combined at the conclusion of the acetic acid demonstration. The recovered metals stream consisted primarily of lead bullets, copper jackets, and pebbles. The metals were placed in nine 55-gallon drums for off-site recycling. This material was accepted for recycling by the smelter, although the site was charged a fee.

☐ Precipitate Sludge: The precipitate tank used for removing flocculated lead species from the spent leachant was emptied on two occasions. The precipitates were removed by pumping the sludge from the bottom of the precipitation tank into 55-gallon drums.

Table 7-4. Residuals Disposal for the Vendor 1 Demonstration

Process Stream	Stream Description	Average Lead Concentration (mg/Kg)	Hazardous/ Nonhazardous	Disposal Method	Mass of Stream (kg)[a]	Unit Cost ($)	Total Cost ($)
T	Processed soil	722[b]	Hazardous	Landfill	146,058	$0.30	$43,496
T	Processed soil	122	Nonhazardous	Returned to range	29,937	$0.00	$0
P[c]	Precipitate sludge	11,990	Hazardous	Landfill	19,731	$1.41	$27,782
Q_{(1)}	Process solution	328 mg/L	Hazardous	Landfill	39,000 gal	$1.25	$48,750
Q_{(2)}	Pad runoff	< 5 mg/L	Nonhazardous	Discharged to POTW	60,000 gal	$0.00	$0
Z	Organic matter	6,457	Hazardous	Landfill	1,240[d]	$0.77	$958
M	Jig concentrate	484[b]	Hazardous	Landfill	4,082	$1.53	$6,260
Metals	Particulate metals	239,000	Hazardous	Recycled	4,715[e]	$0.53	$2,495

(a) Total mass of process streams is on a wet weight basis.
(b) This material was classified as hazardous waste because the TCLP analyses for lead were greater than 5.0 mg/L.
(c) Mass of material in this stream includes the spent bag filters.
   (d) Mass of material in this stream was estimated to be 1% of the total raw soil processed; moisture content was approximately 85%.
(e) Mass of material in this stream was estimated from the weights of the drums reported by the off-site recycling facility.

During the first removal of sludge, twenty-one 55-gallon drums were filled with material. The second removal of sludge was done during demobilization and resulted in the filling of twenty-seven 55-gallon drums. These drums were sent off site for hazardous waste disposal in a landfill. The bag filters that were used to further remove lead precipitate from the regenerated leachant were placed in five 55-gallon drums and sent off site for hazardous waste disposal in a landfill.

❑ Organic Matter: Organic matter from the range soil was separated by a screen attached to the blade mill. The material (with very high moisture content) that collected on the organic's screen was placed in six 55-gallon drums for storage until the end of the demonstration, at which time it was disposed of as hazardous waste in a landfill.

❑ Process Solutions: Process solutions (mostly leachant) from the acetic acid process continually leaked from various places in the plant onto the pad and into the containment pond. During operations, Vendor 1 was instructed to reuse the solution in the containment pond by pumping it back into their system. On multiple occasions, however, approximately 60,000 gallons of pad runoff was discharged to the sewer via a drainpipe attached to the containment pond. The discharge to the sewer was authorized only after the solution in the containment pond was analyzed for lead content and pH, and if the potential for further inclement weather existed. If the content was below 5 mg/L lead and at or near neutral pH, it was deemed as safe to discharge. At the end of Vendor 1's demonstration, samples of the process solutions remaining in the process equipment were neutralized as part of a testing procedure to examine whether the solution could be discharged to the POTW. These tests resulted in an exothermic reaction and the formation of a gel-like substance, which eventually broke down. The remaining process solution was disposed of off site as hazardous waste in a landfill.

❑ Other Residuals: PPE, such as gloves and coveralls. The PPE and other miscellaneous spent supplies were placed in a 55-gallon drum. The drum was sent off site for disposal in a hazardous waste landfill.

7.1.3 Vendor 1 Plant Reliability

In general, plant reliability was low, and this caused excessive downtime and a progressive deterioration in the quality of the processed soil. Table F-3 in Appendix F contains the daily information on plant operations logged by Battelle. The raw soil feed belt operation was the main determinant of process uptime. In the 24 days available for soil processing, the plant was operational for a total of 139.5 hr, or 65% of the available time. During operation, there was no indication that the plant, which was designed for continuous operation, managed to reach steady state as far as material flow through the various process elements was concerned. Various process elements often could not be operated in tandem because equipment and sizing limitations caused material to accumulate at intermediate points, thereby causing difficulties in estimating the material balance of the system. Mobilization of the plant took 14 days (not including transportation to the site) and demobilization took 10 days.

The following difficulties were encountered by Vendor 1 during the demonstration:

☐ Inadequate Feed Control: The feed hopper was prone to bridging and was often inoperable without manual intervention. An operator had to continually knock soil down onto the plate feeder. The plate feeder also repeatedly broke down, causing more downtime. The feed belt conveyor was sometimes manually loaded by dropping the soil on it with a backhoe whenever the feed hopper was down.

☐ Insufficient Soil Deagglomeration: Initially, the acetic acid process plant was designed to use the blade mill as the principal slurrying mechanism. During mobilization, the vendor determined that the blade mill did not sufficiently deagglomerate clayey soils, and, in fact, created clay soil balls which rolled off the screen decks and onto the processed soil conveyor before the leachable lead could be removed. A dual-cell attrition scrubber was ordered and installed on September 7, causing mobilization to be extended by 3 days. The dual-cell scrubber did provide sufficient attrition and was used for the rest of the demonstration.

☐ Undercapacity of the Processed Coarse Soil Dewatering Mechanism: During the initial validation test, the vendor determined that the sand screw basin volume was insufficient to provide adequate coarse soil dewatering. The volume of the sand screw basin was increased 50% by cutting and welding larger sheet metal onto the screw basin. This modification caused approximately 12 hours of system downtime.

☐ Undercapacity of the Processed Fine Soil Dewatering Mechanism: The vacuum belt filter that was brought to the site for dewatering the processed fines was undersized for the material encountered. After 2 weeks of processing, and many hours of process downtime while the dewatering of the processed fine soil caught up with the rest of the operation, it was decided that a plate-and-frame press would be ordered and installed to dewater the fines. The plate-and-frame press was installed on October 1. The smaller vacuum filter was then employed for additional dewatering of the coarse processed soil. After these modifications it appeared that sufficient soil dewatering was achieved.

☐ Clogging of Filter Cloths: The plate-and-frame and vacuum belt presses were equipped with filter cloths. The cloths were used to retain the processed soil so that it would be pressed and dewatered. A precoat or filter aid, such as diatomaceous earth, was not applied to the filter cloth. This resulted in the plugging of the filter cloth of both the plate-and-frame and vacuum belt filters. The filter cloths were removed and disposed of as nonhazardous waste. New filter cloths were installed on October 9, and lasted until the end of the demonstration. The replacement of the filter cloths resulted in approximately 20 hours of downtime.

☐ Inadequate Neutralization of Excess Acid in the Processed Soil: The excess acid leachant in the processed soil was not always sufficiently neutralized because there was no efficient mechanism to introduce the lime slurry in a controlled manner, nor was there a mechanism to mix it with the processed soil. The inability to neutralize the excess acid, coupled with the inadequacy of the dewatering mechanism, sometimes created hazardous

working conditions due to acetic acid and vapors. Sampling personnel were forced to switch from Level D to Level C PPE (respirator and rubber boots) on September 28, to sample the processed soil.

☐ Bag Filter Installation: Bag filters were added after the clarifying tanks to remove any remaining precipitated lead particles. The addition caused a 4-hour delay in operation. The bag filters were replaced daily, and the spent filters were placed in 55-gallon drums.

Part of the difficulty encountered by Vendor 1 may be attributed to insufficient preparation time caused by an aggressive schedule. Vendor 2 not only had the benefit of a longer preparation period, but Vendor 2 also was in a position to learn from some of the difficulties encountered by Vendor 1. However, the following observations can be made with respect to the difficulties encountered by Vendor 1:

☐ From Vendor 1's bench-scale testing report, the precipitation process does not appear to have been investigated adequately at the bench-scale level. The inability to precipitate and separate lead from the spend leachant and to return a relatively clean leachant to the process resulted in increasing levels of lead in the processed soil. Inadequate precipitation could have resulted from a number of causes. The proprietary precipitant used by the vendor, ThioRed®, is reported by the manufacturer (ETUS, Inc. Marketing Information) as working best at a pH of 8.5. No pH adjustment was done by the vendor during precipitation, which appeared to have been conducted at a pH of 3.2. to 3.6. Even if the precipitant is claimed to work over a wide pH range, there was no apparent bench-scale basis or field process control to ensure that enough precipitant was being added. The presence of significant levels of other metals, such as iron in the leachant, could have interfered with the reaction. The manufacturer also recommends using ThioRed® only as a polishing step after other precipitants have removed most of the metal. All these conditions need to be determined during bench-scale testing, as mentioned in Section 2.3.2.

☐ Materials handling arrangements proved to be insufficient. Some essential pieces of equipment, such as a device for mixing lime slurry into the processed soil or a device for dewatering the precipitate sludge, were missing from the outset of the demonstration. Other equipment, such as the vacuum belt filter, were inadequately sized for the type of feed material encountered. Even if the berm has been adequately characterized, the processing plant should be slightly oversized to accommodate inevitable variability in the texture (particle size) and contamination level of the feed soil at small-arms ranges. During this demonstration, the somewhat higher-than-expected fine soil fraction overloaded the capacity of the leaching module creating significantly lower feed rate and many hours of operational downtime.

☐ Process control was insufficient. For the most part, process parameters such as sludge levels, the pH level in tanks, contact times, and throughput rates could not be adequately determined. It was unclear how Vendor 1 planned to monitor many of these parameters at the desired locations in the plant. The chief plant operator could not verify the content of lead in the regenerated leachant in time to make a difference.

☐ Adequate staffing of the plant was not implemented, perhaps due to budgetary constraints. Although the plant operators appeared knowledgeable in running the plant, the use of additional operators, more automation, and an on-site process chemist with appropriate measurement devices would have given plant operators better control over the process and facilitated on-site decision making and appropriate responses.

7.1.4 Removal of Other Metals

In addition to lead, the process removed significant amounts of other metals of concern from the soil. As seen in Table 7-5, levels of copper, zinc, and antimony were reduced significantly. Just as for lead, however, removal could have been greater if the leachant had been properly regenerated.

7.1.5 Reusability of the Processed Soil

The physical condition of the processed soil appeared to be suitable for return to the active berm, in that the material had a loose, granular structure that could absorb the impact of bullets. However, inadequate dewatering, presence of leachable levels of lead, and low pH made the processed soil unsuitable for reuse in the berm. There was concern that lead could continue to leach out of the soil, especially under its low-pH condition. The processed soil that did not pass TCLP was disposed of off site as hazardous waste.

7.2 PERFORMANCE OF VENDOR 2 AND THE HYDROCHLORIC ACID PLANT

The process streams that were sampled to evaluate Vendor 2's plant performance are described in Figures 5-8 and 5-9 in Section 5. Appendix G contains the detailed analytical data for the samples collected during Vendor 2's demonstration.

7.2.1 Lead Removal by Vendor 2 Process

The total lead removal by the hydrochloric acid process averaged 96% on a daily basis as shown in Table 7-6. An 80% confidence interval shows total lead removal from 4,117 ±435 mg/kg in the raw berm soil (U) to 165 ±15 mg/kg in the processed soil (T), well below the design goal of 500 mg/kg. Leachable lead in the processed soil was consistently below the targeted 5-mg/L TCLP level. The relatively low standard deviations and the relatively narrow confidence intervals indicate that steady state was reached and maintained.

Table 7-7 shows the lead assays of various process streams. The pH ranges of the process streams are also given in Table 7-7. Most of the lead in the raw soil was concentrated at the front end of the plant in the form of large metal fragments that were manually removed from the jig bed. The metal particulates thus removed had a lead assay of over 49%. Additional ionic lead was concentrated in the precipitate sludge, which had an assay of almost 2%. The organic matter that was screened out of the overflow from the first sand screw had an assay of over 1% lead, but was not significant in mass.

Table 7-5. Removal of Metals of Concern by the Vendor 1 Process

Date	Total Copper Result			TCLP Copper Result	
	Raw Soil (mg/kg)	Processed Soil (mg/kg)	Removal (%)	Raw Soil (mg/L)	Processed Soil (mg/L)
15-Sep	812	59.8	92.6	0.754	0.768
21-Sep	1,516	99.0	93.5	1.00	1.78
25-Sep	1,525	215	85.9	0.736	7.01
2-Oct	1,317	359	72.7	0.562	7.08
4-Oct[a]	99.0 - 215	165	N/A	0.736 - 1.00	5.14
10-Oct	1,943	797	59.0	3.30	10.9
12-Oct	1,943	729	62.5	3.30	21.3

Date	Total Zinc Result			TCLP Zinc Result	
	Raw Soil (mg/kg)	Processed Soil (mg/kg)	Removal (%)	Raw Soil (mg/L)	Processed Soil (mg/L)
15-Sep	72.2	16.9	76.6	0.49	1.07
21-Sep	168	18.7	88.9	0.442	0.662
25-Sep	127	32.2	74.7	0.448	2.46
2-Oct	103	45.4	55.9	0.293	1.96
4-Oct[a]	18.7 - 32.2	22.7	N/A	0.442 - 0.448	0.925
10-Oct	159	65.0	59.0	0.572	2.29
12-Oct	159	88.1	44.5	0.572	3.31

Date	Total Antimony Result			TCLP Antimony Result	
	Raw Soil (mg/kg)	Processed Soil (mg/kg)	Removal (%)	Raw Soil (mg/L)	Processed Soil (mg/L)
15-Sep	105	31.7	69.69	0.325	0.141
21-Sep	89.3	44.1	50.62	0.132	0.067
25-Sep	180	54.5	69.72	0.233	0.012
2-Oct	139	91.8	33.96	0.670	0.057
4-Oct[a]	44.1 - 54.5	64.2	N/A	0.132 - 0.233	0.066
10-Oct	219	171	21.92	0.671	0.142
12-Oct	219	261	-19.18	0.671	0.143

(a) The soil processed during this time period was reprocessed soil from past TCLP failures.

The feed soil was split into coarse (K) and fine (L) fractions in the first sand screw. The coarse fraction, containing the metal fragments, was processed through the jig to bring its lead assay down from approximately 806 mg/kg (K) to approximately 175 mg/kg (C). The lead removed from the coarse fraction was collected manually from the jig bed. The fine fraction was processed in the leaching module, which reduced the lead concentration from 417 mg/kg (L) to approximately 163 mg/kg (F). The lead removed from the fine fraction went into the leachate (Q_f), which had an assay of approximately 96 mg/L of lead. Precipitation with sodium hydroxide and polymeric flocculant was used to bring the lead content in the regenerated leachant (Q_c) down by almost 90% to 11.5 mg/L of lead.

Good pH control was maintained during the entire processing. An on-site process chemist constantly checked pH and lead assay at key points in the process. The pH in the precipitation

**Table 7-6. Overall Removal of Total and Leachable Lead with the
Hydrochloric Acid Process**

Date	Total Lead Raw Soil (mg/kg)	Total Lead Processed Soil (mg/kg)	Total Lead Removal (%)	TCLP Lead Raw Soil (mg/L)	TCLP Lead Processed Soil (mg/L)
15-Nov	4,819	143	97	18.4	3.07
16-Nov	4,819	178	96	18.4	1.83
20-Nov	4,152	125	97	20.7	0.958
21-Nov	3,567	134	96	37.3	1.32
22-Nov	4,068	115	97	33.5	0.56
23-Nov	5,194	232	96	31.9	1.75
25-Nov	5,194	235	95	31.9	2.15
26-Nov	5,040	181	96	36.3	1.97
27-Nov	5,040	165	97	36.3	2.84
29-Nov	5,040	230	95	36.3	3.44
30-Nov	3,351	233	93	40.4	2.53
2-Dec	3,351	177	95	40.4	1.85
3-Dec	3,351	132	96	40.4	1.36
4-Dec	2,743	113	96	13.7	2.35
5-Dec	2,743	127	95	13.7	3.06
6-Dec	2,743	123	96	13.7	0.757
Statistics					
n	8	16	16	8	16
Avg.	4,117	165	96	29	2.0
Std. Dev.	869	46	1.0	10	0.86
80% C.I.	4,117±435	165±15	96±0.34	29±5.0	2.0±0.29

C.I. = confidence interval.
n = number of independent measurements.

Table 7-7. Stream Assays of Lead in the Hydrochloric Acid Process

Process Stream	Stream Description	pH Range	Total Lead Result (mg/kg) Nov. 22-26	Total Lead Result (mg/kg) Dec. 2-3	Total Lead Result (mg/kg) Dec. 4-6	TCLP Lead Result (mg/L) Nov. 22-26	TCLP Lead Result (mg/L) Dec. 2-3	TCLP Lead Result (mg/L) Dec. 4-6	Avg. Total Lead Conc. (mg/kg)
U	Raw soil	5-5.5	4,068	3,351	2,743	33.5	40.4	13.7	4,117
T	Processed soil	4.7-9.6	115	177	127	0.56	1.85	3.06	165
M	Jig concentrate	N/A	1,644	N/A	N/A	36.6	N/A	N/A	1,644
P	Precipitate sludge	7.7	16,455	N/A	21,571	1,474	N/A	2,235	19,013
Z	Organic matter	N/A	NA	N/A	10,896	N/A	N/A	7.84	10,896
Metals	Particulate metal	N/A	491,900	N/A	N/A	N/A	N/A	N/A	491,900
C	Coarse processed fraction	5.6	135	N/A	214	4.41	N/A	44.2	175
F	Fine processed fraction	N/A	N/A	175	150	N/A	2.78	1.95	163
K	Feed to jig	N/A	360	N/A	1,251	13.9	N/A	64.5	806
L	Leach circuit feed	N/A	N/A	428	405	N/A	9.98	11.9	417
Q_f [a]	Spent leachant	1.5-1.6	103	N/A	88.2	N/A	N/A	N/A	95.6
Q_c [a]	Regenerated leachant	1.4-1.5	7.66	N/A	15.3	N/A	N/A	N/A	11.5

(a) Leachant concentration measured in units of mg/L.
N/A = Not applicable/available.

process was maintained above 7.0, as had been determined during bench-scale tests. The regenerated leachant subsequently was returned to a pH of 1.4 to 1.5.

The coarse processed soil (C) sample collected on December 5 showed an unexpectedly high TCLP lead concentration (44.2 mg/L in Table 7-7) that is inconsistent with the low result for the final processed soil (T) collected on the same day. The final processed soil (T) is a combination of coarse (C) and fine (F) processed fractions. Upon further scrutiny, this high value for C appeared to be a result of a high value measured for only one of the six replicates analyzed and averaged for this sample. This single high measurement could be the result of an isolated metal particle in the coarse fraction.

Table 7-8 shows the estimated mass flow of soil material and lead through the various process streams. The average total lead concentration numbers were reported by the laboratory on a dry-weight basis. Therefore, the wet weights measured for each process stream were adjusted for moisture content.

Table 7-8. Mass Distribution of Lead in Various Process Streams for Vendor 2

Process Stream	Stream Description	Moisture Content (%)	Mass of Process Stream (kg) [a]	Average Lead Concentration (mg/kg)	Mass of Lead (kg)	Mass Percentage of Lead (%) [b]
U	raw soil	9.1	757,507	4,117[c]	2,836	100
T	processed soil	22.8	745,968	165	95	3.4
P	precipitate sludge	62.9	26,672	19,013	188	6.6
Z	organic matter	40.0	800[d]	10,896	5.2	0.2
Metals[e]	jig bed metals	5.0[e]	7,859[f]	491,900[g]	3,673	129.5[h]

(a) Total mass of process streams are on a wet weight basis.
(b) Overall balance equation : U = T + P + Z + Metals.
(c) Concentration of total lead in the raw soil varied considerably from day-to-day.
(d) Mass of material in this stream was estimated to be 1 % of the total feed.
(e) This stream contained particulate metals collected from the jig bed and a small amount of soil; moisture content was assumed to be 5 %.
(f) Mass of material in this stream was estimated from the weights of the drums reported by the off-site recycling facility.
(g) Concentration of lead in the recovered metals stream was measured by pyrometallurgical analysis conducted on three samples collected from this stream.
(h) This number has the highest uncertainty because of the high variability of this stream and the limitations of the analytical methods.

Of the overall lead present in the raw feed soil, about 6% was recovered in the precipitate sludge. Approximately 3% of the lead remained in the process soil that was returned to the range. Most of the lead in the original feed soil was recovered in the jig bed rather than in the jig concentrate (M). The jig concentrate was returned to the sand screw. The mass balance is skewed mainly because of the uncertainty in the jig bed metals concentration number (491,900 mg/kg). This concentration was estimated by analyzing three grab samples of the large bullet and gravel fragments by special pyrometallurgical techniques. The mass percentages of lead in the other streams (U, T, P, and Z) are more reliable estimates.

Although most of the lead appears to be removable by coarse separation processes, such as screening or hand-sorting, the additional 6% removed in the leaching module enabled the processed soil (T) to meet TCLP criteria. Reblending the processed coarse and fine fractions may have also helped the final processed soil (T) pass TCLP by redistributing the leachable lead from the coarse to the fine fraction, in which it is better sequestered during the leaching test. This was conjectured to be the case during the bench-scale treatability tests conducted by Vendor 2.

7.2.2 Vendor 2 Process Residuals

Residuals generated during the processing performed by Vendor 2 are shown in Table 7-9 and include the following:

Table 7-9. Residuals Disposal for the Vendor 2 Demonstration

Process Stream	Stream Description	Average Lead Concentration (mg/kg)	Hazardous/ Nonhazardous	Disposal Method	Mass of Stream (kg)[a]	Unit Cost ($)	Total Cost ($)
T	Processed soil	165	Nonhazardous	Returned to range	745,968	$0.00	$0
P	Precipitate sludge	19,013	Hazardous	Recycled	26,672	$0.18	$4,850
Q_c	Process solution	< 5 mg/L[b]	Nonhazardous	Discharged to POTW	22,000 gal	$0.00	$0
Z	Organic matter	10,896	Hazardous	Reblended with process soil[c]	800[d]	$0.00	$0
Metals	Particulate metals	491,900	Hazardous	Recycled	7,859[e]	$0.53	$4,158

(a) Total mass of process streams are on a wet weight basis.

(b) The lead concentration measured during the demonstration was 11 mg/L. The final concentration measured prior to discharge was less than 5 mg/L due to additional processing of the discharge by the vendor and the influx of rainwater into the containment pond.

(c) In the future, this stream should be reblended with the precipitate sludge, or disposed of as hazardous waste.

(d) The mass of material in this stream was estimated to be 1% of the total feed.

(e) Mass of material in this stream was estimated from the weights of the drums reported by the off-site recycling facility.

❑ Recovered Metals: Metal particulates recovered from the plant processing were stored in 55-gallon drums until the end of the demonstration. Most of the particulate metal was recovered from the jig manually by panning and hand-sorting the bed. The recovered metals consisted primarily of lead bullets and copper jackets. The metals were placed in fifteen 55-gallon drums for off-site recycling.

❑ Precipitate Sludge: The precipitation tank used for removing flocculated lead species from the spend leachant was emptied on a daily basis into the sludge storage tanks. The precipitate sludge was then pumped to the thickener tank where diatomaceous earth was added as a bulking agent. From this tank, the sludge was pumped to the plate-and-frame filter press. The press removed excess moisture from the sludge and formed a filter cake, which had moisture content of 56%. The filter cake that was generated during processes was stored in three roll-off bins. The total mass of filter cake was 27,720 pounds.

Because the filter cake contained a high total metals (lead, copper, zinc, antimony) assay of almost 11%, including 2% lead, the roll-off bins were sent off site for recycling.

☐ **Organic Matter:** During processing, organic matter from the range soil was removed by a screen that processed the overflow from the attrition scrubber. The material that collected on the organic screen was stored until the end of the demonstration, at which time it was reblended with the processed soil that had been returned to the range. Approximately 800 pounds of organic matter was generated during processing. The organic matter had a moisture content of approximately 40%. This procedure is not recommended at future sites because, as seen in Table 7-7, the organic matter (Z) had a lead assay of almost 1% and TCLP lead of 7.84 mg/L, making it a hazardous stream. The organic matter may be better blended with the precipitate sludge that is sent for off-site disposal.

☐ **Process Solutions:** Process solutions from the hydrochloric acid process occasionally leaked into the containment pond. As was the case for Vendor 1, Vendor 2 was instructed to reuse the solution and rainwater in the containment pond by pumping it back into the system. No discharges to the POTW were made during processing operations. The only discharge to the POTW occurred at the conclusion of the demonstration, and this was authorized only after the solution in the containment pond was analyzed for lead content and pH. Because the lead content of the water in the pond was below 5 mg/L and at or near neutral pH, it was deemed as safe to discharge. Approximately 22,000 gallons of water containing less than 5 mg/L lead was discharged to the POTW.

☐ **Other Residuals:** As with Vendor 1, PPE, such as gloves and coveralls, was stored in 30-gallon polyethylene drums until analysis from air monitoring was performed. The air monitoring analyses from the second demonstration indicated that no significant residual lead dust was present during the demonstration processing. Therefore, the PPE and other miscellaneous spent supplies were placed in a garbage disposal container. The container was emptied via sanitation truck on a biweekly basis.

7.2.3 Vendor 2 Plant Reliability

The above discussion of Vendor 2's plant performance results show that the processing criteria were met or exceeded. For this plant, downtime was less than 2% of the total operational time. The process was shut down for 2 hours for minor adjustments during 162 hours of available processing time (see Table G-3 in Appendix G). Plant mobilization (not including transportation time to the site) was completed in 14 days and demobilization was completed in 10 days. Although Vendor 2 had more preparation time and the benefit of learning from the difficulties encountered by Vendor 1, the following factors also contributed to smooth operation:

☐ **Comprehensive Bench-Scale Treatability Testing:** Bench-scale testing conducted by Vendor 2 addressed all aspects of the process, including selection of a suitable precipitant and operating conditions.

☐ **Knowledge of Soil Characteristics and Contaminant Distribution:** The soil characteristics (particle size distribution, moisture content, etc.) and contaminant distribution (in the

various size fractions) were adequately measured by Vendor 2 during the bench-scale tests. All reagent (leachant, precipitant, coagulant, etc.) and process kinetics consider-ations (solid-liquid ratios, contact times, pH, etc.) were established during the bench tests.

☐ Material Handling Considerations in the Design: Scale-up was done with detailed atten-tion to material handling aspects. For wet processing of this type, material handling problems are, in general, the primary cause of downtime.

☐ Adequate Field Process Control: The process parameters designed during bench-scale treatability testing and pilot plant scale-up appeared to have been achieved by the plant during the field operation. Steady state was reached early in the operation and was maintained on a daily basis throughout the demonstration by building adequate process control in the design and by constant operator attention. Parameters such as pH, solid-liquid ratios, contact time, and lead concentration in the leachate were maintained at the desired levels. The processing rate averaged 6.3 tons/hr over the 18 days of operation compared to the design goal of 10 tons/hr. However, the processing rate appeared to be limited by the availability of soil storage space for the processed soil awaiting analytical verification; the plant was oversized and appeared to have some excess capacity to reach higher feed rates. The processing rate did not vary much during day-to-day operations and any variations were intentional.

☐ Plant Flexibility: The processing plant was designed in a modular fashion. This allowed equipment operators flexibility in handling the minor equipment changes necessitated by the variability in feed composition. This was important during processing because the feed material varied over time. The proportions of fine and coarse fractions, as well as the amounts of particulate metal contained in the raw soil delivered to the operations pad, changed daily. This happens because the berm itself often contains soil acquired from various sources during previous range maintenance operations. During this demonstra-tion, Vendor 2 made the following plant modifications:

- The screen and coarse material jig were removed from the plant because a higher proportion of clay in the feed soil caused clay balls to form on the screen during validation testing and subsequently entered the coarse material treatment train. The size separation was instead effected in the first sand screw. Elimination of the screen and coarse material jig resulted in oversize and coarse metal fragments reaching the fines jig, which had a 1/8-inch slotted punch plate. This jig was not of a type suited to handle the coarse metals, which were retained on the punch plate. The coarse metal fragments retained on top of the jig bed were removed and hand-sorted by an operator at the end of each day.

- Sand screw modification caused some downtime during the initial plant startup. The angular pitch of the first sand screw was elevated to obtain the correct discharge heights and clearances. The modification took about 1 day. This modification was done on the day that analytical verification results of the validation pilot test were received.

– During operations, slight equipment modifications were made to fix the centrifuge, replace the jig bed pump motor, and fix a pipe leak on the attrition scrubber. These modifications caused a total downtime of about 2 hours

7.2.4 Removal of Other Metals

Table 7-10 shows, that in addition to lead, other metals of concern were removed during processing. Copper, the most prevalent metal after lead, was reduced in the soil by 97%. Also, 89% of the total zinc and 60% of the total antimony in the berm soil were removed.

7.2.5 Reusability of the Processed Soil

The soil processed by Vendor 2 was returned to the active berm. The processed soil was loose textured and suitable for absorbing bullet impacts. The processed soil appeared to be chemically suitable as well. Heavy metals had been significantly removed, the soil passed the TCLP test, and the pH had been restored. BDM applied three types of grass seeds to the processed soil returned to the berm. Indications are that revegetation is progressing as desired.

Table 7-10. Removal of Metals of Concern by the Vendor 2 Process

Date	Total Copper Result			TCLP Copper Result	
	Raw soil (mg/kg)	Processed Soil (mg/kg)	Removal (%)	Raw Soil (mg/L)	Processed Soil (mg/L)
15-Nov	2,302	50.0	97.8	1.12	0.768
16-Nov	2,302	48.6	97.9	1.12	0.164
20-Nov	1,958	54.0	97.2	1.10	0.080
21-Nov	1,659	60.3	96.4	1.16	0.155
22-Nov	1,975	63.0	96.8	0.913	0.022
23-Nov	2,456	70.7	97.1	0.790	0.005
25-Nov	2,456	81.0	96.7	0.790	0.000
26-Nov	2,461	51.5	97.9	0.854	0.000
27-Nov	2,461	63.1	97.4	0.854	0.197
29-Nov	2,461	85.3	96.5	0.854	0.455
30-Nov	1,612	62.5	96.1	0.676	0.367
2-Dec	1,612	53.3	96.7	0.676	0.106
3-Dec	1,612	48.1	97.0	0.676	0.095
4-Dec	1,329	54.2	95.9	2.02	0.330
5-Dec	1,329	58.0	95.6	2.02	0.118
6-Dec	1,329	50.0	96.2	2.02	0.061
Statistics					
n	8	16	16	8	16
Avg.	1,969	59.6	96.8	1.08	0.183
Std. Dev.	418	11.2	0.701	0.417	0.207

Table 7-10. Removal of Metals of Concern by the Vendor 2 Process (cont.)

Date	Total Zinc Result			TCLP Zinc Result	
	Raw soil (mg/kg)	Processed Soil (mg/kg)	Removal (%)	Raw Soil (mg/L)	Processed Soil (mg/L)
15-Nov	182	17.6	90.3	0.348	1.07
16-Nov	182	14.3	92.1	0.348	0.166
20-Nov	158	17.0	89.2	0.33	0.087
21-Nov	136	18.5	86.4	0.389	0.435
22-Nov	157	21.2	86.5	0.367	0.145
23-Nov	193	19.6	89.8	0.246	0.402
25-Nov	193	23.0	88.1	0.246	0.059
26-Nov	190	14.8	92.2	0.379	0.131
27-Nov	190	16.4	91.4	0.379	0.192
29-Nov	190	21.9	88.5	0.379	0.227
30-Nov	127	14.8	88.3	0.359	0.220
2-Dec	127	13.4	89.4	0.359	0.038
3-Dec	127	14.1	88.9	0.359	0.091
4-Dec	111	15.2	86.3	0.275	0.156
5-Dec	111	16.2	85.4	0.275	0.161
6-Dec	111	17.0	84.7	0.275	0.119
Statistics					
n	8	16	16	8	16
Avg.	157	17.2	88.6	0.337	0.231
Std. Dev.	30.4	2.93	2.30	0.051	0.249

Date	Total Antimony Result			TCLP Antimony Result	
	Raw soil (mg/kg)	Processed Soil (mg/kg)	Removal (%)	Raw Soil (mg/L)	Processed Soil (mg/L)
15-Nov	255	56.1	78.0	0.154	0.140
16-Nov	255	64.5	74.7	0.154	0.369
20-Nov	216	54.0	75.0	0.098	0.340
21-Nov	190	80.3	57.7	0.188	0.485
22-Nov	206	89.0	56.7	0.179	0.677
23-Nov	262	105	59.9	0.080	0.575
25-Nov	262	115	56.1	0.080	1.11
26-Nov	248	73.6	70.3	0.405	0.483
27-Nov	248	77.8	68.6	0.405	0.137
29-Nov	248	128	48.5	0.405	0.212
30-Nov	172	93.5	45.6	0.906	0.041
2-Dec	172	65.5	61.9	0.906	0.433
3-Dec	172	68.6	60.1	0.906	0.306
4-Dec	149	65.0	56.4	0.157	0.147
5-Dec	149	77.4	48.1	0.157	0.256
6-Dec	149	89.0	40.3	0.157	0.551
Statistics					
n	8	16	16	8	16
Avg.	212	0.391	59.9	0.271	0.391
Std. Dev.	41.0	0.264	11.2	0.275	0.264

7.3 QUALITY ASSURANCE (QA)

Sampling and analysis were conducted under the QA procedures described in the Technology Demonstration Plan (Battelle, 1996). Appendices F and G contain the QA data related to the evaluation of the demonstration.

7.3.1 Field Sample Collection

The biggest QA challenge in the demonstration was obtaining representative samples of hetero-geneous process streams, especially when particulate metals were present. Process streams at the front-end of the process plant, such as the raw soil (U), jig concentrate (M), and oversize fraction (O), were likely to contain particulate metals. Collecting representative samples from the matrix containing particulate metal contaminants is made difficult by what is known in the mining industry as the "nugget" effect. Depending on whether a particle or fragment of metal is col-lected in the sample or not, the analytical result for the sample can vary widely. The larger the metal particle size, the greater the nonreproducibility of analytical results. In a previous field effort at an ammunition manufacturing site, where 2-gram aliquots collected from the raw soil were analyzed, variability between replicate analysis (as measured by the relative standard deviation or RSD) was over 50% (Fristad et al., 1996). For the Fort Polk evaluation, an ambi-tious attempt was made, in consultation with mining industry experts, to keep the RSD down to around ±25%.

In this evaluation, large composite samples, as much as 300 lb for the raw soil, were collected and processed to maintain sampling representativeness, as described in Section 6.3. The final processed soil (T) is generally not expected to have any particulate metals, because these should have been removed in the physical separation circuit. However, because of the possibility that some particulate metals may have persisted, larger composites were collected for T in all cases except for the October 12-13 sample. This last T sample represented additional processing that was negotiated between Vendor 1 and the site support contractor after the sample preparation equipment had been demobilized. However, as shown in Section 7.3.2.1, sufficiently large grab samples were collected and processed even for this T sample so that the variability (RSD) was maintained below 25%, probably due to the absence of any large metal particles in the processed soil.

7.3.2 Sample Preparation and Analysis

Sample preparation (drying, grinding, splitting) ensured that the maximum particle size in the composite sample was successively reduced, so that the large composite (~200 to 300 lb) could be reduced to smaller and smaller representative aliquots (2 to 8 grams) that were amenable to analysis by laboratory instrumentation (see Figure 6-2 in Section 6 for sample preparation scheme).

For total metals analysis, each composite sample was successively ground in the field until it passed through a 10-mesh screen. After the sample was ground to 10 mesh it was split in the same manner as the TCLP samples, and 1-L sample containers were filled and shipped overnight

to Battelle's Laboratory in Columbus, Ohio. There, the samples were ground into two fractions, a -10 + 30 mesh fraction and a -200 mesh fraction. Representative 8-gram samples of the -200 mesh fraction were split and analyzed. Even at a maximum metal particle size of 200-mesh (clay size), the representative aliquot size of 8 grams, is much larger than the 2 grams recommended by the EPA method for digestion by ICP. Special modifications were made to the microwave digestion procedure to accommodate 8-gram instead of 2-gram samples. As many as four 8-gram replicates were analyzed and averaged for each composite sample collected to obtain a representative analysis. The final total metals result for any sample included the following elements:

☐ For many samples, there was a small fraction that just could not be ground below 30 mesh by the laboratory grinder (ball mill) in any reasonable amount of time. In such cases, the +30 mesh fraction was separately digested in its entirety and analyzed.

☐ For many raw berm soil samples, malleable metals such as lead and copper flattened out during grinding and were recovered as part of a +10-mesh fraction recovered during subsequent sieving. Three samples of the +10-mesh fraction (mainly metals) were analyzed separately by special pyrometallurgical techniques.

☐ The final total metals result for a given sample, therefore, consisted of the weighted average of the total metals concentrations in the three fractions: +10 mesh, +30 mesh, and -200 mesh.

To collect "representative" aliquots for TCLP analysis, each composite sample was dried and ground, if necessary, until it passed through a 3/8-inch sieve as suggested in the EPA standard method. The dry sample was then run through a sample splitter several times until 1-L sample containers could be filled and shipped overnight to Battelle's laboratory in Columbus, Ohio. When the samples were received by the laboratory, they were immediately split into 100-gram aliquots for the TCLP digestion process. It should be noted that, in a sense, the TCLP test is never really representative of this type of material because when the maximum expected metal particle size in the sample is 3/8-inch, the aliquot size extracted would have to be at least 42 lb (see Table 6-1). To overcome this difficulty, multiple 100-gram aliquots were collected by splitting each large composite sample to average out any variability due to the potential presence of any particulate metal in an individual sample. For Vendor 1, as many as six TCLP replicate analyses were conducted on each sample. The number of replicate TCLP analyses per sample was reduced to four for Vendor 2, after the variability between replicates was found to be lower than originally expected because of the extensive sample preparation procedure. The final reported TCLP value for each composite was the average of the six (or four) replicates.

7.3.2.1 Sample Preparation and Analytical Precision

The precision or reproducibility of the sample preparation and analysis as measured by the RSD for the total lead measurements is shown in Figure 7-1. The RSD is calculated as the standard deviation between replicate analytical measurements divided by the average of the measurements. For total lead, RSDs for both processed and raw soil preparation and analysis were below
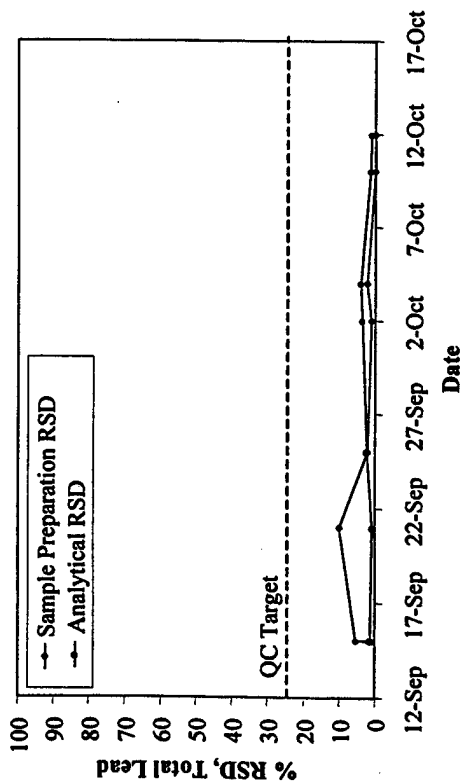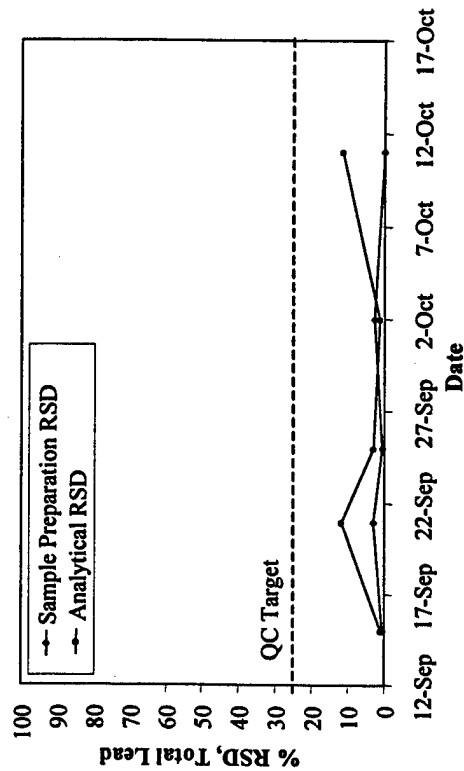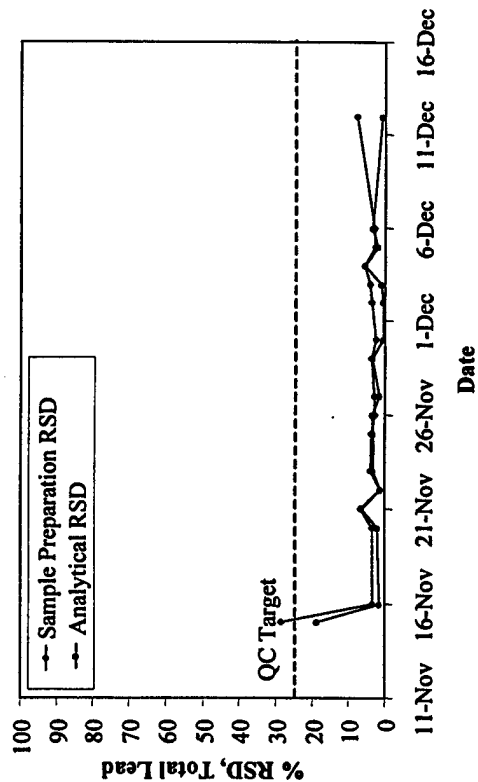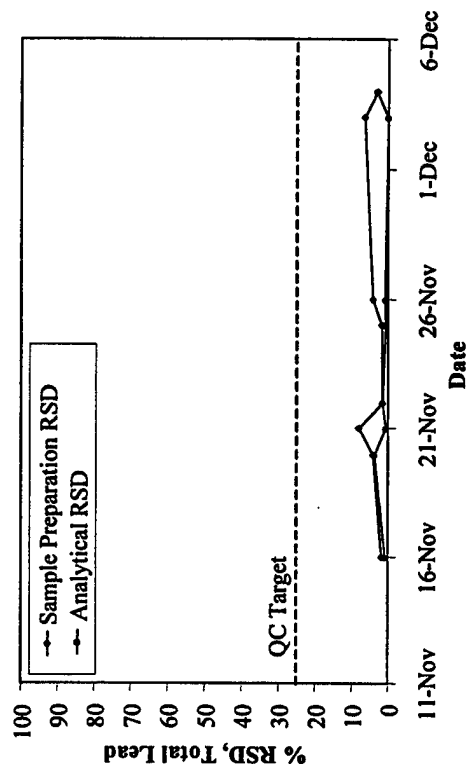
Figure 7-1. Precision Evaluation for Total Lead Analysis

25%. The sample preparation RSD represents the reproducibility between the two 1-L bottles of soil collected for each sample. The analytical RSD represents the reproducibility between duplicate analyses performed on the soil in the same 1-L bottle. On November 16, the processed soil sample from Vendor 2's operation had a sample preparation RSD that was slightly above the 25% target.

Precision of the TCLP lead analysis (see Figure 7-2) was lower because of the inherently unrepresentative nature of the TCLP test, as discussed above. The TCLP analysis of the processed soil had better precision than the TCLP analysis of the raw soil. The raw soil contains more particulate metals and this accounts for the variability. Although the target RSD of 25% was exceeded on some samples, in general, the occasional high variability was averaged out by analyzing multiple replicates and averaging them to provide a level of confidence agreed to by all parties before the demonstration.

7.3.2.2 Field Blanks

Two field blanks, consisting of clean sand, were processed through all the sample collection and preparation equipment, one during Vendor 1's demonstration and one during Vendor 2's demonstration. The objective was to see if there was any cross-contamination during sampling. As shown in Table 7-11, the field blanks did not show any significant levels of the metal analytes, compared with the concentrations of these metals in the raw and processed soil samples.

7.3.2.3 Accuracy of the Analysis

Matrix spikes were analyzed with a minimum frequency of 5% (one for each batch of 20 samples). Because samples often arrived in smaller batches, this frequency was exceeded for the demonstration.

Figure 7-3 shows the accuracy of the analysis as measured by the percent matrix spike recovery in the processed soil samples. The analyte was spiked into the extract for TCLP analysis and into the soil samples prior to digestion for total lead analysis, as required by the respective EPA standard methods. Matrix spike recoveries were mostly within the targeted range of 75% to 125%. Two of the matrix spike recoveries (on the third and eighth batches) for total lead during Vendor 1's processing were significantly higher than targeted. The occasional over recoveries are probably due to isolated metal particles in the single aliquots spiked to measure the recovery. Because four replicates were analyzed and averaged for each regular (unspiked) sample, any isolated variations should have been averaged out.

Lead was the main focus of the demonstrations, and analytical instrumentation and procedures were aligned to obtain maximum accuracy and precision for lead analysis. The QA data for other metals and other matrices are included in Appendices F and G. In general, the accuracy and precision of the copper and zinc analyses were within an acceptable range. The analytical procedure was modified at the beginning of the demonstration by adding hydrochloric acid to the nitric acid digestion to improve antimony recovery. Low matrix spike recoveries were seen for antimony due to the fact that the sample concentrations were near or below the quantitation limit for antimony.

(a) Processed Soil - Vendor 1
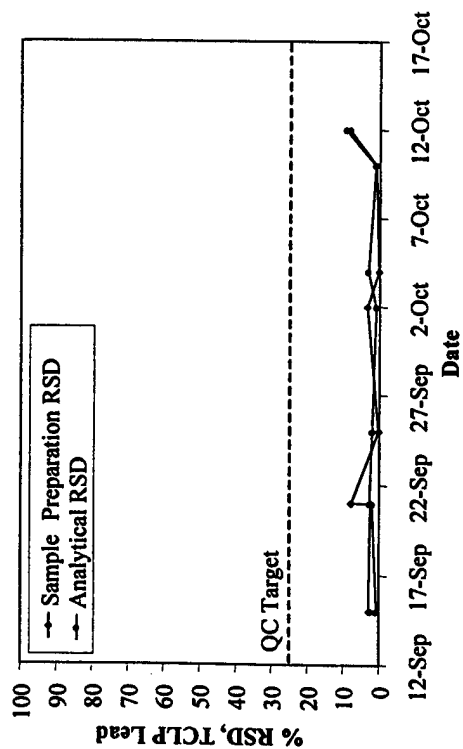
(b) Raw Soil - Vendor 1
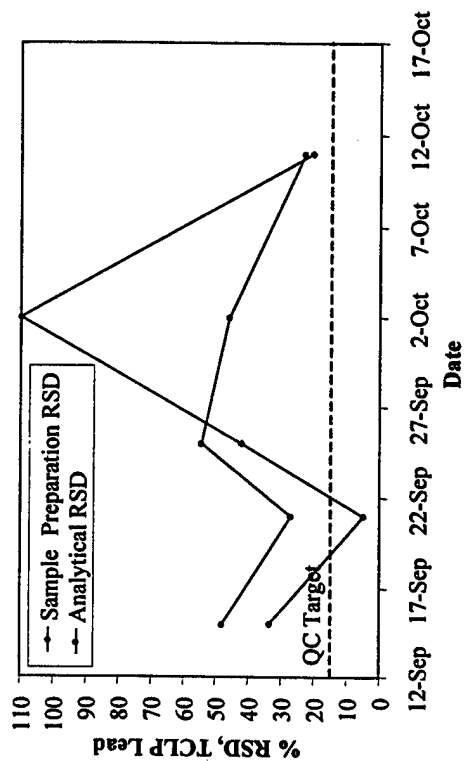
(c) Processed Soil - Vendor 2
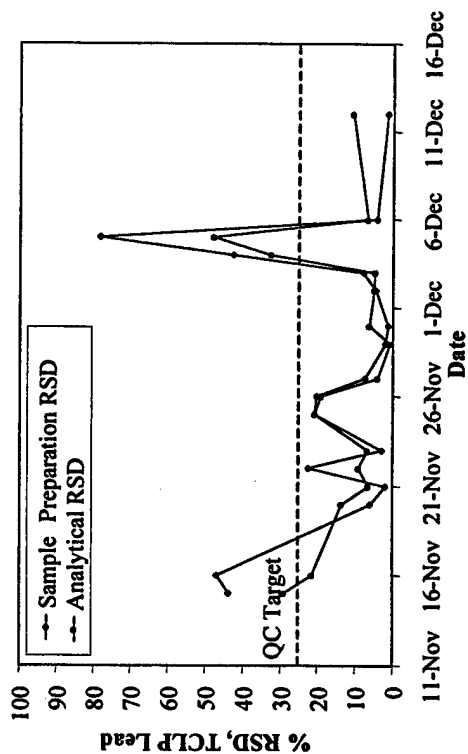
(d) Raw Soil - Vendor 2
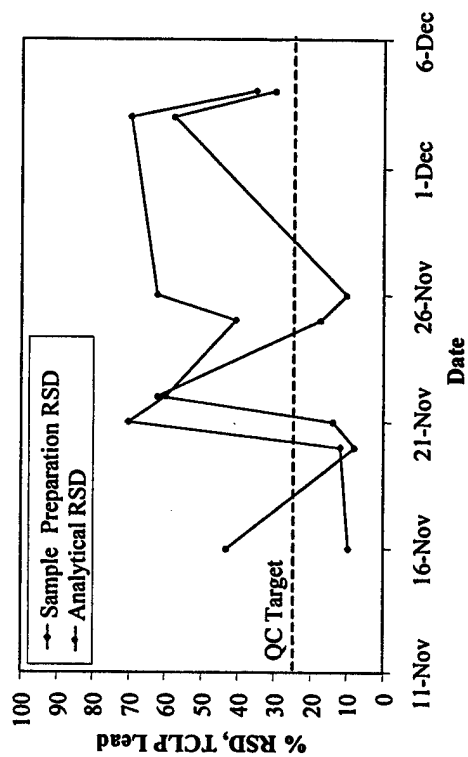
Figure 7-2. Precision Evaluation for TCLP Lead Analysis

Table 7-11. Field Blanks Processed During the Soil Washing Demonstrations

Field Blank Sampling Date	Blank Matrix	Lead Result (mg/kg)	Copper Result (mg/kg)	Zinc Result (mg/kg)	Antimony Result (mg/kg)
October 5	Sand	2.70	11.60	7.29	0.31
December 3	Sand	6.58	6.19	6.25	1.09

7.3.2.4 Calibration Checks and Method Blanks

Instruments were calibrated daily by running initial and continuing calibration checks. The recoveries for initial calibration and check standards were between 85 and 115%, indicating that the ICP instrument was within calibration during the analysis. Figure 7-4 shows the results of the method blanks that were run frequently to ensure the absence of analytical interferences. Except for one slightly elevated reading, all the blanks were well below the method detection limit (MDL). The one slightly elevated blank is well below the lowest total metal value measured in any of the samples in the demonstration.

Given the speed with which the analysis had to be completed (2- to 3-day turnaround times for TCLP results were implemented so that the soil could be moved out of the limited storage space on the demonstration pad), the data quality is relatively consistent. Compared with the poor reproducibility reported in many previous studies with particulate metals-contaminated soils (Fristad et al., 1996), the present data set had better precision and accuracy that allow meaningful interpretation of results.

7.4 COST PERFORMANCE

For the cost performance evaluation of the two separation/leaching technologies, the costs during site setup, mobilization, operation, and demobilization were categorized as either fixed costs or variable costs. Fixed costs are incurred irrespective of the amount of soil processed. Examples of fixed cost items are environmental assessment, regulatory permitting, site characterization, bench-scale treatability testing, engineering and administration, site preparation, process plant lease (vendor), transportation, mobilization, and demobilization. Variable costs are dependent on the total amount of soil processed. Variable costs in the two demonstrations included costs for chemicals used, utilities (power and water) required, operating labor, sampling and analysis, consumables and supplies, soil excavation and hauling, and residual disposal. Any recycled metals recovered from processing were considered as a credit to the residual disposal variable cost. Appendix I contains details of the cost estimating basis used. For estimation purposes, equipment (depreciation) cost is assumed to be fixed, because the vendors plan to depreciate the equipment used for the demonstration by a fixed amount at each site.

7.4.1 Vendor 1 Process Cost

Table 7-12 shows the costs incurred during the demonstration of the acetic acid process. The site preparation and sampling/analysis costs are included in this table. Because of frequent downtime
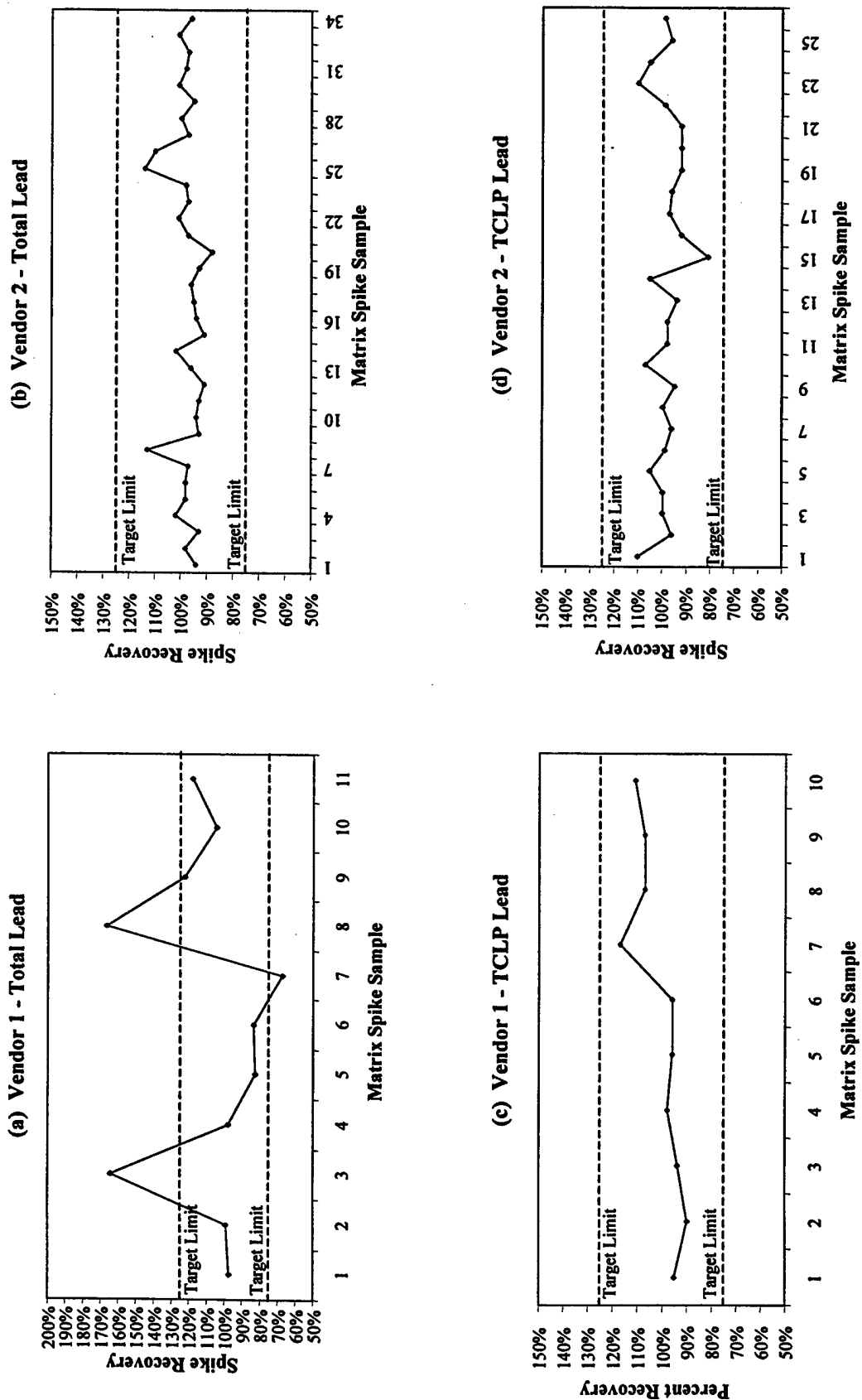
Figure 7-3. Accuracy Evaluation of Lead Analysis of Processed Soil

(a) Vendor 1 - Total Lead

(b) Vendor 2 - Total Lead

(c) Vendor 1 - TCLP Lead

(d) Vendor 2 - TCLP Lead
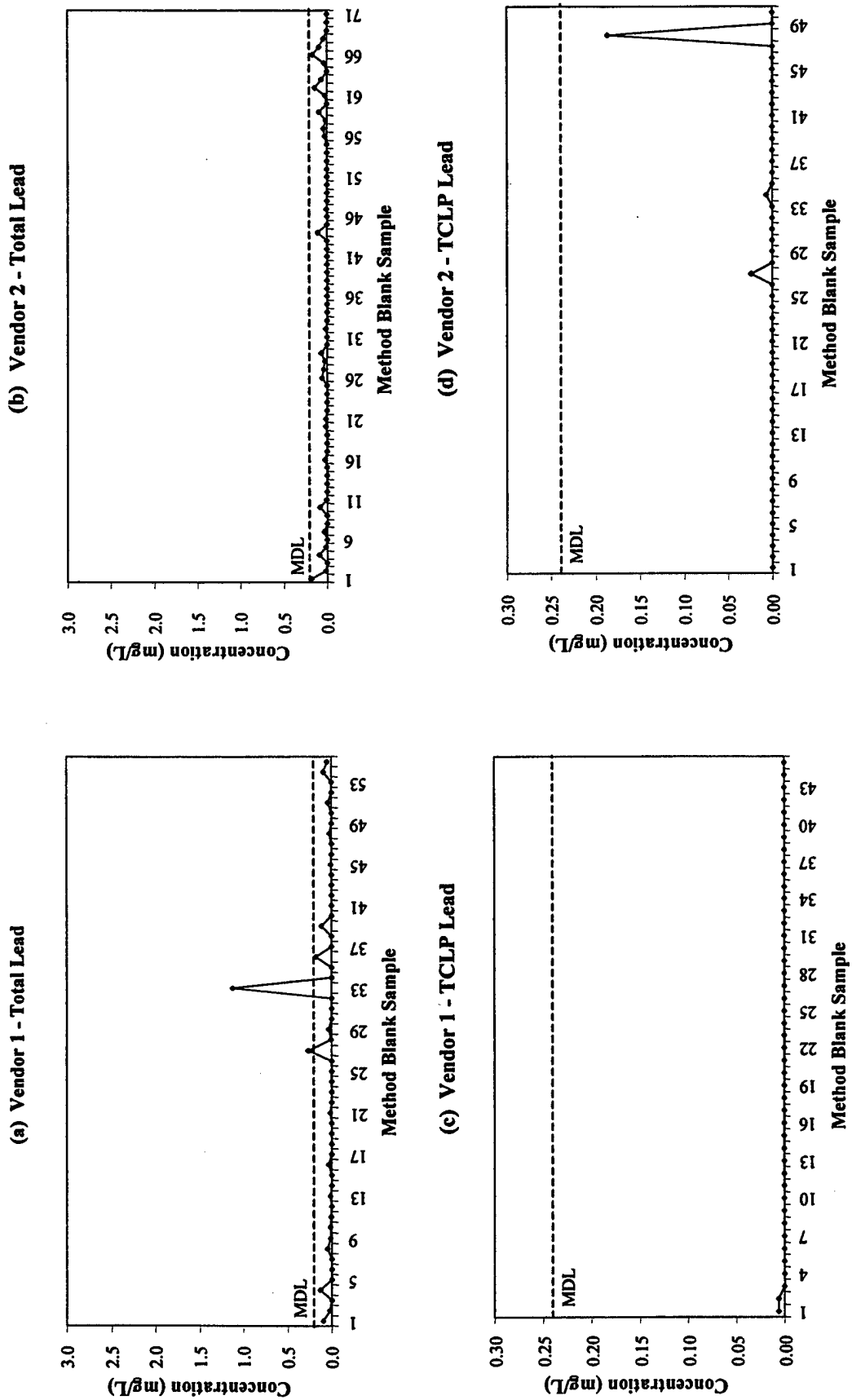
Figure 7-4. Method Blanks Analyzed for the Fort Polk Demonstration

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and the inability to meet processing targets, the costs in Table 7-12 are not really representative of relatively smooth processing that results in acceptable processed soil quality. For example, every process component used was flooded with excess acetic acid in an attempt to remove leachable lead. In addition, because the processed soil was not dewatered properly, a considerable amount of acetic acid solution was retained by the processed soil and removed from

Table 7-12. Costs Incurred During the Acetic Acid Demonstration

Item	Basis	Demonstration Costs 263 tons
Fixed Costs		
Permitting and Regulatory (Site)	NEPA, HASP, & other permitting	$73,199
Site Characterization (Site)	Planning, sampling, and analyses	$56,171
Vendor Selection (Site)	Selection and contracting, plan preparation	$135,686
Bench-Scale Testing (Vendor)	1 representative sample	$17,739
Site Preparation and Support (Site)	Pad construction and accessory rentals	$150,839
Engineering and Administrative (Vendor)	Administrative and assessment	$45,000
Transportation (Vendor)	Plant and personnel mobilization	$79,200
On-Site Mobilization (Vendor)	Equipment procurement and shakedown	$63,000
Equipment (Vendor)	25% depreciation over 4 cleanups	$105,800
Decontamination and Demobilization (Vendor)	Disassembly, decontamination, and demobilization	$47,600
Total - Fixed Costs		**$774,234**
Variable Costs		
Site Excavation & Hauling (Vendor)	Backhoe equipment, excavation & hauling	$12,419
Labor (Site)	1 site superintendent for 420 hours	$25,200
	1 health and safety officer for 420 hours	$21,000
Utilities (Site)	Electricity, 5,000 kWh/month @ $0.075/kWh	$750
	Water, 51,250 gal @ $8.07/kgal	$414
	Phone, $220/month	$440
Labor (Vendor)	1 supervisor for 420 hours	$119,800
	1 operator for 420 hours	
	1 safety officer for 420 hours	
	4 technicians for 420 hours each	
Chemicals (Vendor)	Acetic acid, 9,415 gal @ $5.17/gal	$48,635
	ThioRed®, 1,210 gal @ $10.91/gal	$13,201
	Diatomaceous earth, 0 lb @ $0.53/lb	$0
	Flocculant, 110 gal @ $3.31/gal	$364
	Hydrated lime, 2,000 lb @ $0.40/lb	$800
Consumables / Supplies (Vendor)	PPE, gloves, tarps, accessories	$10,394
Sampling & Analyses (Site)	Accessories, other equipment rentals	$19,983
- Labor (Site)	1 supervisor for 420 hours	$25,200
	2 technicians for 420 hours each	$25,200
- Analyses (Site)	219, sample prep & TCLP analysis	$50,000
	233, sample prep & total metals analysis	
Residuals, Waste Shipping/Handling (Vendor)	Bulk solid waste & recovered metals credit	$80,991
Effluent Treatment (Site)	Wastewater, 19,000 gal @ $1.25/gal	$48,750
Total - Variable Costs		**$503,541**
Total - Project Costs		**$1,277,775**
Total - Cost/ton of soil processed		**$4,858**

the plant. This required a higher acid usage rate to make up for the lost acetic acid. The numerous leaks from the process also resulted in higher chemical and utility costs; however, the reuse of the containment pond water alleviated some of these additional costs. Therefore, the chemical and utility costs that were incurred on site during the first demonstration are probably higher than for a routine range maintenance operation.

Operating labor was used inefficiently during the demonstration because of the prolonged downtime spent in changing, replacing, or adjusting the processing equipment. Sampling and analyses costs were higher than normal for this demonstration because of the special sample collection and preparation procedures used and because the limited storage space on the pad for the processed soil required extremely fast turnaround time for the TCLP analysis. Collecting multiple grab samples of the processed soil (as described in Section 6-3) could reduce sampling and analytical costs if desired by site representatives. The residuals disposal costs were extraordinarily high for the vendor demonstration. The disposal costs for this demonstration included the hazardous waste disposal of the following residuals:

- Fifty-two 55-gallon drums of precipitate sludge
- 161 tons of processed soil that failed TCLP
- Five 55-gallon drums of organic matter
- Six 55-gallon drums of jig concentrate
- 26,000 gallons of spent process solution
- Nine 55-gallon drums of oversize material containing particulate metals
- Five 55-gallon drums of spent bag filters
- One 55-gallon drum of used PPE
- 20,000 gallons of contaminated stormwater runoff.

7.4.2 Vendor 2 Process Cost

The hydrochloric acid process costs incurred during the second demonstration provided a better basis for projecting the costs for a full-scale processing operation. Table 7-13 shows the costs incurred during the Fort Polk demonstration and the projected costs for a normal range processing operation for the same volume of range soil with similar soil characteristics and similar processing targets. The total cost for the range maintenance demonstration at Fort Polk that processed 835 tons of berm soil was around $1.17M, at an average cost of around $1,400/ton. Minor adjustments were made to the second column of costs in Table 7-13 to account for the differences between the requirements for a demonstration (which involves more plant operator involvement, sampling, and analysis) versus those for a routine range maintenance or remediation. However, these elements did not affect the total or unit cost considerably. Fixed costs accounted for two-thirds of the total cost of the range maintenance and therefore the unit cost (per ton) remains relatively high. At larger sites, the unit cost per ton of soil processed by hydrochloric acid is expected to be much lower.

Residuals disposal costs for the second vendor totaled $9,008. This figure is slightly lower than may be expected at other sites. At Fort Polk, the vendor was not required to dispose of organic residue matter which contained significant lead. For this demonstration, the organic matter was

Table 7-13. Incurred and Routine Costs of the Hydrochloric Acid Demonstration

Item	Basis	Demonstration Costs 835 tons	Normal Processing Costs 835 tons
Fixed Costs			
Permitting and Regulatory (Site)	NEPA, HASP, & other permitting	$73,199	$73,199
Site Characterization (Site)	Planning, sampling, and analyses	$56,171	$56,171
Vendor Selection (Site)	Selection and contracting, plan preparation	$135,686	$135,686
Bench-Scale Testing (Vendor)	1 representative sample	$17,739	$17,739
Site Preparation & Support (Site)	Pad construction and accessory rentals	$150,839	$150,839
Engineering & Administrative (Vendor)	Administrative and assessment	$41,571	$41,571
Transportation (Vendor)	Plant and personnel mobilization	$173,692	$173,692
On-site Mobilization (Vendor)	Equipment procurement and shakedown	$23,825	$23,825
Equipment (Vendor)	25% depreciation over 4 cleanups	$233,075	$233,075
Decontamination and Demobilization (Vendor)	Disassembly, decontamination, and demobilization	$20,000	$20,000
Total - Fixed Costs		**$925,797**	**$925,797**
Variable Costs			
Soil Excavation/Hauling (Vendor)	Backhoe equipment, excavation/hauling	$12,419	$12,419
Labor (Site)	1 site superintendent for 300 hours	$18,000	$18,000
	1 health and safety officer for 300 hours	$15,000	$15,000
Utilities (Site)	Electricity, 5,000 kWh/month @ $0.075/kWh	$750	$750
	Water, 49,300 gal @ $8.07/kgal	$398	$398
	Phone, $220/month	$440	$440
Labor (Vendor)	1 supervisor for 300 hours	$51,845	$34,563
	2 engineers for 300 hours each[a]		
	1 chemist for 300 hours		
	5 technicians for 300 hours each[a]		
Chemicals (Vendor)	HCl acid, 5,200 gal @ $0.60/gal	$3,141	$3,141
	NaOH, 5,850 gal @ $0.60/gal	$3,517	$3,517
	Diatomaceous earth, 11,300 lb @$0.53/lb	$6,044	$6,044
	Flocculant, 1,000 gal @ $3.31/gal	$3,311	$3,311
	Hydrated lime, 1,275 lb @ $0.40/lb	$510	$510
Consumables / Supplies (Vendor)	PPE, gloves, tarps, accessories	$8,235	$8,235
Sampling & Analyses (Site)	Accessories, other equipment rentals	$19,983	$16,383
- Labor (Site)	1 supervisor for 300 hours	$18,000	$18,000
	2 technicians for 300 hours each[a]	$18,000	$9,000
- Analyses (Site)	240, sample prep & TCLP analyses	$57,000	$14,280
	529, sample prep & total metals analysis		
Residuals, Waste Shipping/Handling (Vendor)	Bulk solid waste & recovered metals credit	$9,008	$9,200
Effluent Treatment (Site)	Wastewater, 0 gal @ $1.25/gal[a]	$0	$27,500
Total - Variable Costs		**$245,601**	**$200,693**
Total - Project Costs		**$1,171,398**	**$1,126,490**
Total - Cost/ton of soil		**$1,402**	**$1,349**

(a) These costs have been changed for the normal maintenance estimate.

simply blended with the processed soil. For routine or full-scale operations at other sites, the organic matter should instead be blended with the precipitate sludge or be disposed of separately as hazardous waste. The spent process solution, which also contained significant amounts of lead, may have to be disposed of as hazardous waste unless lead levels can be reduced so that it can be discharged to the local POTW.

Because some small-arms range sites have as much as 10,000 tons of soil or more, a cost projection for a hydrochloric acid remediation of that size is shown in Table 7-14. It is assumed that the same size plant (20-tons/hr quoted capacity) will be used for the normal processing of sites up to 10,000 tons. Inherent in this assumption is the projection that the performance of the processing plant will be maintained at a higher throughput rate of 20 tons/hr. The maximum throughput rate measured during the second demonstration was about 7.5 tons/hr, but the processing rate was limited more by the availability of storage space for the processed soil awaiting verification than by the processing equipment capacity. Implicit in the scale-up and routine cost projections is the assumption that the plant will be required to meet similar processing targets (5 mg/L TCLP lead and 500 mg/kg of total lead). The projected unit cost for normal processing or the remediation of 10,000 tons of contaminated berm soil is around $170/ton. This projection does not take into account any profit or fee that the vendor may charge. It is only meant to serve as a reasonable estimate for the incurred costs, should a full-scale small-arms range maintenance action or remediation be undertaken.

An additional cost consideration when using hydrochloric acid for leaching is its corrosive effect on equipment. Analysis of the precipitate sludge (December 6, 1996, sample) showed that this sludge contained over 4% iron. Although some of this iron may have leached out of the soil or bullets, it is possible that some of it was leached out of the carbon steel equipment itself. Use of hydrochloric acid at a very low pH (below 2) may reduce the life of the equipment. Alternatively, the plant may have to utilize more expensive stainless steel equipment, which will increase capital costs.

At other sites, the following factors could affect the cost elements in Table 7-13:

❑ Type of Soil. Higher fines content will reduce processing speed.

❑ Lead Concentration and Particle Size Distribution. High lead concentrations will not necessarily increase costs if most of the lead occurs in the larger particle size range. However, higher concentrations of lead fines and molecular species may increase processing requirements.

❑ Speciation of Lead and Other Heavy Metals. At sites where lead and other heavy metals are mostly in the form of carbonates, processing costs will be similar to those at Fort Polk. Other heavy metal species may require additional processing.

❑ Processing Targets. More stringent targets will require additional processing and cost. On the other hand, range maintenance activities may not have any regulatory-driven processing targets.

Section 8.2.7 provides a comparison of the costs of common alternatives to separation/leaching.

Table 7-14. Scale-up Costs of the Hydrochloric Acid Process

Item	Basis	Scale-Up Costs 10,000 tons
Fixed Costs		
Permitting and Regulatory (Site)	NEPA, HASP, other permitting	$73,199
Site Characterization (Site)	Planning, sampling, and analyses	$56,171
Vendor Selection (Site)	Selection and contracting	$135,686
Bench-Scale Treatability Tests (Vendor)	1 representative sample	$17,739
Site Preparation and Support (Site)	Pad construction and accessory rentals	$150,839
Engineering and Administrative (Vendor)	Administrative and assessment	$41,571
Transportation (Vendor)	Plant and personnel mobilization	$173,692
On-site Mobilization (Vendor)	Equipment procurement and shakedown	$23,825
Equipment (Vendor)	25% depreciation over 4 cleanups	$233,075
Decontamination and Demobilization (Vendor)	Disassembly, decontamination and demobilization	$20,000
Total - Fixed Costs		**$925,727**
Variable Costs		
Site Excavation / Hauling (Vendor)	Backhoe equipment, excavation & hauling	$124,190
Labor (Site)	1 Superintendent/HSO for 480 hours	$28,800
Utilities (Site)	Electricity, 5,000 kWh/month @ $0.075/kWh	$1,125
	Water, 80,000 gal @ $8.07/kgal	$646
	Phone, $220/month	$660
Labor (Vendor)	1 supervisor for 480 hours	$134,400
	1 engineer for 480 hours each	
	1 chemist for 480 hours	
	3 technicians for 480 hours each	
Chemicals (Vendor)	HCl acid, 62,275 gal @ $0.35/lb	$21,796
	NaOH, 70,060 gal @ $0.44/lb	$30,826
	Diatomaceous earth, 50 tons @ $800/ton	$40,000
	Flocculant, 7,200 gal @ $2.20/gal	$26,347
	Hydrated lime, 8 tons @ $89/ton	$712
Consumables / Supplies (Vendor)	PPE, gloves, tarps, accessories	$50,994
Sampling & Analyses (Site)	Accessories, other equipment rentals	$34,873
- Labor (Site)	1 supervisor for 480 hours	$28,800
	1 technician for 480 hours	$14,400
- Analyses (Site)	360, sample prep & TCLP analysis	$86,040
	800, sample prep & total metals analysis	
Residuals, Waste Shipping / Handling (Vendor)	Bulk solid waste & recovered metals credit	$110,180
Effluent Treatment (Site)	Wastewater, 22,000 gal @ $1.25/gal	$27,500
Total - Variable Costs		**$726,289**
Total - Project Costs		**$1,688,086**
Total - Cost/ton of soil processed		**$168**

8. Comparison with Competing Technologies

This section provides a comparison of physical separation and acid leaching with potentially competing technologies. The comparison follows the two-stage screening approach applied in technology screening for RCRA corrective action and Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) remedial actions. A variety of reference documents are available if more detailed technology performance and selection data are needed (Conner, 1990; U.S. EPA, 1991, EPA/540/2-91/014; U.S. EPA, 1992, EPA/540/S-92/011; U.S. EPA, 1995, EPA/540/R-95/512).

8.1 TECHNOLOGY REVIEW AND PRESCREENING

This section provides overviews of two alternative remedial action technologies that can be applied to remediate metal contamination in small-arms range soils. The following paragraphs also describe the basis for selecting a small group of candidate alternatives to receive more detailed evaluation in the remediation and/or maintenance of small-arms ranges. A discussion of a broader range of technologies that can be applied to remediate small-arms range soils is given in Appendix E.

8.1.1 Secure Landfill Disposal

For disposal at an offsite landfill, the contaminated material is first excavated and then transported to an approved facility. This off-site disposal can reduce human health risk by reducing the risk for direct contact with the contaminated material. An engineered landfill reduces the migration of the contaminants by using liners and caps.

Depending on the type of waste and the contaminant levels, pretreatment may be required before landfill disposal. RCRA wastes subject to land ban restrictions must be treated to meet required standards prior to disposal (40 CFR Part 268).

Hazardous wastes typically are sorted according to their compatibility and then placed below ground in control cells. Control cells usually are separated by an earthen berm or other separation method. Each control cell is constructed by placing and compacting about 2 to 3 feet of waste in a double-lined earthen pit. A 1-ft-thick layer of cover soil is placed over the wastes at the end of each day. Waste is added to approximately 16 to 20 feet above grade. Hazardous waste landfills are designed to control leachate, gaseous emissions, runoff and drainage, and settling. Data needs for the selection of disposal at a landfill depend on permitting requirements of the landfill but probably would include the following:

☐ Contaminant type and concentration
☐ TCLP test
☐ Volume of waste.

Cost estimates for excavation and disposal at a RCRA landfill range from $280 to $500/yd³ (U.S. EPA, 1992, EPA/540/S-92/011).

8.1.2 Ex Situ Solidification/Stabilization (done on site)

Solidification/stabilization (S/S) treatment changes the physical and chemical characteristics of the waste to reduce contaminant mobility (Conner, 1990). Solidification processes lock the waste in a low-permeability matrix. Stabilization chemically converts the waste to a less mobile form. The most widely applied method for S/S treatment of chemical-contaminated waste uses portland cement, portland cement and soluble silicate, lime and fly ash, or a similar silicate-based binder system. Phosphate addition, either with a silicate binder or as the only binding chemical, is not widely reported but can help immobilize lead by forming $Pb_5(PO_4)_3OH$, $Pb_3(PO_4)_2$, and other lead/phosphate compounds with low solubility in neutral to mildly acidic solutions. The contaminated soil must be well mixed to ensure intimate contact with the binder and water. During a curing period, after mixing, the binder reacts with the soil and contaminants to form a less porous, more leach-resistant treated waste form. The treated waste may be particulate or a monolithic solid. Treating waste with cement reduces contaminant mobility by a variety of physical and chemical mechanisms. Much of the immobilization of metals results from precipitation in the elevated pH environment caused by the CaOH released by the cement reactions.

The reported costs for S/S treatment with inorganic binders typically fall in the range from $70/yd³ to $200/yd³. These costs include labor, equipment, monitoring and testing, reagents, and miscellaneous supplies. Not included are costs for equipment mobilization and demobilization, engineering and administration, and health and safety (Arniella and Blythe, 1990; U.S. EPA, 1995, EPA/540/R-95/512). The cost of S/S treatment can be as high as $500/yd³ to $1,000/yd³, if conditions such as small volumes of waste, difficult contaminants such as arsenic, and wet or sticky wastes are encountered (U.S. EPA, 1993, EPA/530/R-93/012). Arsenic could be present in shotfall areas at shotgun ranges, but the arsenic concentrations are unlikely to be high enough to increase the cost of S/S treatment.

8.1.3 Technology Prescreening

S/S treatment with inorganic binders is retained for detailed evaluation and comparison with acid leaching. S/S treatment is widely used for immobilizing metal or other inorganic contaminants in sludge or soil matrices. S/S treatment is an accepted approach for immobilizing metal contaminants in small-arms range soils once the bulk metal constituents have been removed by screening.

Disposal at a permitted landfill is retained for detailed evaluation and comparison with acid leaching. Disposal is the least favored in the hierarchy of waste management methods but is still widely used for managing hazardous waste.

8.2 DETAILED COMPARISON OF ALTERNATIVES

This section provides a discussion of technology alternatives that currently have the best combination of effectiveness, implementability, and cost for typical small-arms range maintenance or remediation projects. The advantages and limitations of these technologies are compared to physical separation and acid leaching. The remedial objectives for processing of small-arms range soils typically will include:

☐ Maximize recovery of lead metal
☐ Reduce total and leachable metals to acceptable levels
☐ Minimize the volume of soil or residuals requiring off-site disposal.

8.2.1 General Applicability of Alternative Technologies to Small Arms Ranges

This section provides descriptions of the operation and performance of the two most favorable alternatives as determined by the technology prescreening:

☐ Excavation and secure disposal at an off-site landfill
☐ Ex situ solidification/stabilization done on site.

The discussion is based on the assumption that both landfill disposal and S/S treatment are preceded by a simple screening operation to remove oversize debris, whole bullets, and large bullet fragments. The debris and bullet metals are transferred to a smelter for lead recovery. The screened soils are either treated with a cement binder at the range or shipped off site to a permitted landfill.

8.2.1.1 Landfill Disposal

Standard construction excavation equipment and methods would be applicable for handling and shipping small-arms range soils for disposal. Conventional methods such as application of water spray or polymer foam during earth moving and placement of plastic sheeting over soil piles would be used to reduce dust releases. The soil would be shipped by dump truck or railcar to an appropriate disposal facility. Truck shipping is more common because it provides greater flexibility for arrival and departure times and fewer loading and transportation logistics. If a terminal is nearby, rail shipping can provide substantial savings, particularly with large-quantity shipments. Unless the terminal is very near to the site, double handling would be needed because trucks are used to move the soil from the site to the terminal where it is then transferred to the railcars. The appropriate permitting for the disposal facility is determined by the RCRA hazardous waste status of the soil. Typical small-arms range soils exhibit excessive lead leachability when subjected to the TCLP resulting in a D008 hazardous waste characteristic, so a RCRA permitted Treatment, Storage, and Disposal (TSD) facility is normally required.

8.2.1.2 Ex Situ Solidification/Stabilization

Ex situ S/S treatment involves excavating the soils, mixing them with binders, and returning the treated waste for use as the core of the impact berm. At some sites, an initial screening step could be used to separate bullets and larger lead fragments from the bulk soil. Treatment of small-arms range soils typically would be accomplished in a mobile treatment plant at the site. Reusing the treated soil to reconstruct the core of the impact berm is applicable only for an active range that does not present an imminent danger to human health and the environment.

S/S treatment generally is not applicable to wastes with volatile organic compounds (VOCs) or high levels of semivolatile organic compounds (SVOCs). If the contaminated material contains organics, mixing operations and heating due to binder hydration may release organic vapors. The asphalt binder used in shotgun target disks (clay birds) is a potential source of polycyclic aromatic hydrocarbons (PAHs) and other SVOCs in shotfall areas of shotgun ranges. Organic analyses should be performed on soils in shotfall areas to determine if the organic content is too high for successful S/S treatment.

Portable batch S/S plants have been developed by vendors incorporating bulk chemical feed and pug mills or other blending equipment. Portable S/S plants are transported via two to six trailers, depending on requirements for upstream processing (crushing, screening), chemical reagent storage, and downstream handling of the stabilized product. Processing rates for large portable plants range from 500 to more than 1,000 tons per day. Pilot-scale plants typically operate at 100 tons per day and may be transported on one trailer.

The S/S-treated product is not necessarily a monolithic block. The binder formulation and mixing method can be designed to produce a granular product and still immobilize the contaminants. The granular material may be appropriate for reuse in the core of the impact berm. In the areas that receive the majority of the bullet impact, a layer of clean soil at least 1 ft thick (NRA, 1989) should be placed over the treated soil.

A variety of performance measures may be applied to S/S-treated waste depending on the selected disposal option, site conditions, and a project-specific risk assessment. Acceptable performance is usually determined by comparing the concentration of contaminant in a leachant, which is a regulatory or risk-based limit. The TCLP test for the D class of leachable hazardous waste characteristic frequently is used, but other tests may apply. It is typical for the total metals content level to be reduced to established requirements at S/S-treated waste sites. For example, in California both the total and leachable metals content requirements apply to all remedial action initiatives.

8.2.2 Evaluation of Short-Term Effectiveness of Alternative Technologies

S/S treatment with an inorganic binder is a commonly accepted method to treat soils containing excessive levels of leachable metals (U.S. EPA, 1993, EPA/530/R-93/012). The S/S treated soils may be reused in the berm of an active range that does not pose an imminent hazard to human health or the environment, as long as the physical texture of the treated soil is granular.

Hardened agglomerates or monoliths would be hard to reuse in an active range, and therefore are undesirable in active range maintenance.

If the range soil cannot be reused on site at an active range, excavation followed by off-site disposal is the most commonly selected remedial option, especially if the amount of soil to be processed is small. A RCRA Part B permit typically would be required for off-site disposal of small-arms range soils that have failed the TCLP. The disposal option reduces the amount of contamination at the site and immobilizes the contaminants in a more secure off-site location but does not reduce the total volume or toxicity of contamination.

The innovative approach of acid leaching reduces the total amount of metal contaminants in the soil. It also allows the recovered metals from the soil washing activity to be recycled by the range owner for reuse. Typically, the metals recovered from the soil washing can be recycled at a smelter at no cost.

8.2.3 Comparison of Long-Term Effectiveness of Alternative Technologies

The long-term effectiveness criterion refers to the ability of an alternative to maintain reliable protection of human health and the environment over time once the cleanup levels have been met. Long-term effectiveness considers the risk posed by treatment residuals and untreated wastes. Factors such as the extent of destruction and reduction of contaminant toxicity, irreversible reduction in contaminant mobility, and reduction in volume of contaminated media are considered.

The physical separation and acid leaching technology provides the best long-term effectiveness by recovering much of the lead and returning it to commercial use. Both the S/S treatment and disposal alternatives rely on chemical and physical containment to immobilize the contaminants. Both of these containment methods have demonstrated effectiveness over periods of years or decades, but effectiveness beyond that time frame cannot be predicted.

8.2.4 Comparison of Toxicity, Mobility, and Volume Reduction of Alternative Technologies

Reductions in toxicity, mobility, and volumes of the contaminants are the three principal measures of the overall performance of a remediation. The 1986 Superfund Amendments and Reauthorization Act (SARA) emphasizes that the preferred alternative should reduce (1) the level of toxicity of contaminants at the site, (2) the spread of contaminants away from the source, and/or (3) the volume or amount of contaminants at the site.

The physical separation and acid leaching technology is the most responsive to the SARA criteria. Leaching reduces the toxicity of the waste by removing contaminants to reach cleanup goals that limit both the total and leachable metal concentrations. S/S treatment reduces the mobility of contaminants to reach cleanup goals that limit the leachable metal concentrations but does not reduce the total metal content. S/S treatment causes the waste volume to increase

because of the added binder. Disposal at a permitted landfill reduces the total metal concentration at the site and immobilizes contaminants in a secure landfill but does not cause a net reduction in the toxicity or volume of contaminated material.

8.2.5 Comparison of Short-Term Effectiveness of Alternative Technologies

Short-term effectiveness refers to the control of adverse impacts on human health and the environment imposed during the construction and implementation of an alternative technology until cleanup goals are achieved. Evaluation of short-term effectiveness should consider potential effects to workers and the general public.

The physical separation and acid leaching technology presents similar types and numbers of potential hazard sources compared to S/S treatment. Both processes require soil transfer and mixing equipment and involve chemical handling and material transfer operations. Depending on the strength and concentration of the acid used, leaching chemicals may present a slightly greater hazard compared to stabilization chemicals such as portland cement or phosphate fertilizer. However, both processes use standard construction and chemical handling equipment.

Off-site disposal significantly reduces the potential hazard to the workers because no processing is required at the range. However, the disposal site may need to perform treatment to meet land disposal restriction standards, so worker hazards are still a potential threat. Off-site disposal also requires transport of the contaminated soil on public streets and highways, which increases the potential for exposure to the general public.

8.2.6 Comparison of Implementability of Alternative Technologies

For each alternative, an assessment must be conducted to judge the suitability of a chosen remedial alternative with respect to the specific capabilities and constraints at the site. The feasibility of implementing a remedial alternative from a technical and administrative standpoint must be determined, and the availability of various goods and services along with monitoring requirements should be considered. Most importantly, the current status and maturity of the treatment must be factored into the implementability.

All of the options use commercial off-the-shelf equipment and technology. S/S treatment and disposal are well-established commercial technologies for small-arms range remediation. A variety of vendors are available to implement either alternative at a range. Application of acid leaching to small-arms range soils is an innovative technology that has not reached full commercial maturity or acceptance. Both S/S treatment and acid leaching require a work area and utilities to support operation of treatment equipment at the site. Disposal requires transportation infrastructure to support vehicle loading and movement of the soil from the range to the disposal facility.

8.2.7 Comparison of Costs of Alternative Technologies

The cost criterion refers to the fixed (capital) cost to design, purchase, and install the remediation option as well as the variable cost of operating and maintaining the option. A detailed cost comparison of fixed and variable costs for landfill disposal, stabilization/solidification, and physical separation/leaching is provided in Table 8-1. The costs used for the alternative technologies chosen are based on figures obtained from the R.S. Means Environmental Restoration Unit Cost Books (R.S. Means, 1996). The detailed cost basis used to generate Table 8-1 can be found in Appendix I. These figures provide reasonably accurate costs for the associated equipment and items used for these types of remedial activities. The costs of the hydrochloric acid full-scale operation have been projected from the Fort Polk demonstration costs incurred for Vendor 2's activity and for site preparation and sampling (see Section 7-4).

Figure 8-1 is a graph of the unit processing cost versus the total soil tonnage processed with the competing technologies. As can be seen from the graph, it is cheaper to utilize landfill disposal when dealing with small sites (2,600 tons or less). S/S-treatment technology (including a preliminary screening step to remove larger metal fragments) becomes more cost effective than landfill disposal at sites larger than 2,600 tons. Hydrochloric acid leaching with physical separation (as conducted by Vendor 2) becomes more cost effective than landfilling at about 5,000 tons. S/S-treatment is cheaper than physical separation/acid leaching regardless of the size of the site. The acetic acid process with physical separation (as conducted by Vendor 1) was not considered in this evaluation because processing difficulties encountered during this demonstration made cost estimation for the scale-up operation difficult.

An off-site technology, such as landfilling, is always cheaper than on-site technologies at smaller sites mainly because on-site technologies have higher fixed costs for site preparation, plant equipment, etc. At larger sites, as the fixed costs are spread out over a larger tonnage of soil processed, on-site technologies become cheaper. Among on-site technologies, stabilization is cheaper than physical separation/acid leaching regardless of the amount of soil processed because stabilization uses simpler equipment and therefore incurs lower capital costs. Residence times of the soil required in stabilization equipment are also lower than those for leaching. This enables much faster processing by stabilization using equivalently sized equipment.

Table 8-1. Cost Comparison of Alternative Technologies

Technology Soil to be Processed	Landfill Disposal Costs 10,000 tons	S/S Costs 10,000 tons	HCl Acid Washing Costs 10,000 tons
Processing Duration	**1 month**	**2 months**	**3 months**
Fixed Costs			
Permitting and Regulatory (Site)	$73,199	$73,199	$73,199
Site Characterization (Site)	$56,171	$56,171	$56,171
Vendor Selection/Contracting (Site)	$25,000	$135,686	$135,686
Bench-Scale Treatability Tests (Vendor)	$0	$17,739	$17,739
Site Preparation and Support (Site)	$15,400	$75,400	$150,839
Engineering and Administrative (Vendor)	$12,000	$41,000	$41,571
Transportation (Vendor)	$52,125	$98,120	$173,692
On-Site Mobilization (Vendor)	$16,500	$22,228	$23,825
Equipment (Vendor)	$55,250	$138,125	$233,075
Decon and Demob (Vendor)	$12,000	$20,000	$20,000
Total - Fixed Costs	$317,645	$677,668	$925,797
Variable Costs			
Site Excavation / Hauling (Vendor)	$1,909,651	$124,190	$124,190
Labor (Site) - Superintendent/HSO[a]	$14,400	$14,400	$28,800
Utilities (Site) - Electricity	$750	$750	$1,125
Utilities (Site) - Water	$323	$4,035	$646
Utilities (Site) - Phone	$440	$440	$660
Labor (Vendor) - Operations Crew	$46,525	$86,600	$134,400
Chemicals (Vendor) - HCl Acid	$0	$0	$21,796
Chemicals (Vendor) - Acetic Acid	$0	$0	$0
Chemicals (Vendor) - ThioRed®	$0	$0	$0
Chemicals (Vendor) - NaOH	$0	$0	$30,563
Chemicals (Vendor) - Cement	$0	$204,897	$0
Chemicals (Vendor) - DE	$0	$18,000	$40,000
Chemicals (Vendor) - Flocculant	$0	$0	$26,347
Chemicals (Vendor) - Lime	$0	$0	$712
Consumables and Supplies (Site)	$12,749	$25,497	$50,994
Sampling and Analyses (Site)	$17,437	$17,437	$34,873
- Labor (Site) - Supervisor	$7,200	$14,400	$28,800
- Labor (Site) - Technician	$3,600	$7,200	$14,400
- Analyses (Site) - TCLP/Totals	$6,480	$42,960	$86,040
Residuals, Waste Shipping and Handling (Vendor)	$0	$87,500	$110,180
Effluent Treatment (Site)	$22,250	$44,500	$27,500
Total - Variable Costs	$2,040,085	$692,806	$762,289
Total - Project Costs	$2,357,730	$1,370,474	$1,688,086
Total - Cost/Ton of Soil Processed	$235	$137	$168

(a) HSO is Health and Safety Officer.
DE is diatomaceous earth.
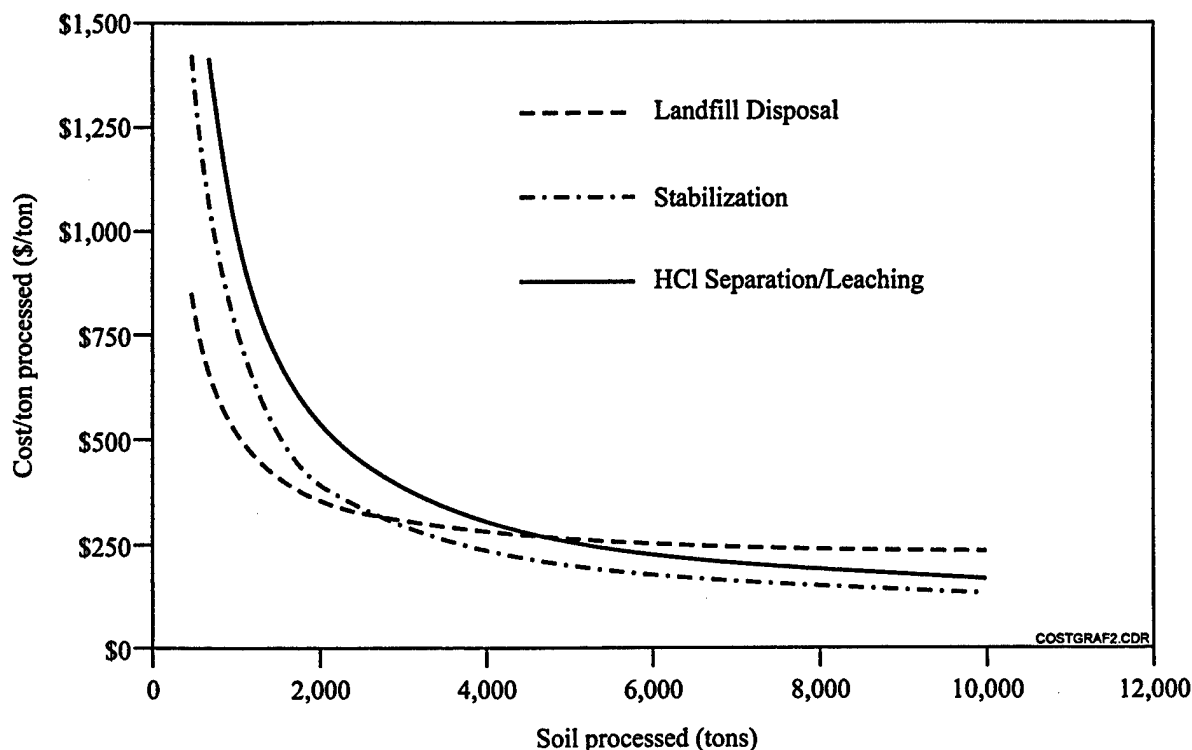
Figure 8-1. Cost Comparison of Competitive Technologies

If a true cost-benefit analysis is undertaken, however, several tangible and intangible benefits of physical separation/acid leaching emerge that may outweigh the cost advantage of landfilling or stabilization irrespective of the amount of soil that requires processing. The following are some of the benefits of physical separation/acid leaching that should be considered by sites trying to identify the best alternative:

❑ With landfilling and stabilization, although the contamination has been immobilized or contained, the liability remains. With physical separation/acid leaching, over 95% of the lead may be removed, recovered, and reused.

❑ Stabilization of an active range may result in a hardened treated material that is physically unsuitable for reuse in the berm. The processed soil from physical separation/acid leaching still retains its loose texture and can be put back in an active berm.

❑ Even at an inactive range, with stabilization and on-site disposal, the site may be put to only limited use, because the contamination remains and is best left undisturbed. On the other hand, if the stabilized material is sent off site for disposal, the cost of this option will increase considerably. With physical separation/acid leaching, the potential uses that the site can be put to increase because most of the contamination is gone.

9. Regulatory Issues

The level of regulatory and administrative oversight depends on whether berm soil processing activities are carried out as range maintenance (at active ranges) or range remediation (at inactive ranges). The following discussion describes the regulatory issues involved at small-arms ranges for the physical separation/acid leaching technology.

9.1 REGULATORY ISSUES AT SMALL-ARMS RANGES

None of the DoD ranges have a cohesive preventive range maintenance program for processing berm soil containing spent bullets and shot. Most efforts have been directed toward safety issues related to clearing unexploded ordnance. Only with matters related to Formerly Used Defense Site (FUDS) and Base Realignment and Closure (BRAC)-driven requirements, influenced by RCRA, have range maintenance and remediation become items of interest. The two principal methods of remediation in the past have been (1) excavation, hauling, and landfilling; and (2) stabilization.

Excavation, hauling, and landfilling can be expensive for larger berms, and this option does not solve the problem on a long-term basis. In fact, it is possible that former owners may become potentially responsible parties (PRPs). Stabilization is more cost effective with regard to capital costs, but it limits the land use for other future beneficial uses. With the massive defense installation realignments occurring now, plus the many installations being closed, the option of stabilization and reuse on site is not a workable solution at many locations, particularly if the range is closing. The physical separation/acid leaching technology has the potential to provide a cost-effective long-term alternative, and is thus expected to be attractive to both site managers and regulators.

From a regulatory standpoint, the principal heavy metal of concern in the range soil is lead. Lead can exhibit RCRA waste characteristics based on toxicity. This is the driver in all the deliberations on the subject of range maintenance or remediation. If the soluble lead concentration in the soil as determined by the TCLP test exceeds the criterion of 5 mg/L, reuse of the land for other beneficial purposes is severely restricted. Range soils contain other metals of concern as well. Copper, zinc, and antimony are present in many types of bullets and are regulated in some states such as California (see Table 9-1).

The maintenance/remediation technology demonstrated in this project involves a combination of physical separation and acid leaching to significantly remove both particulate and ionic lead. This technology has great potential for widespread application throughout the nation. However, even though the TCLP criterion for the processed soil was met, the STLC limit for lead from a Waste Extraction Test (WET) extraction for California hazardous waste designation was not met, as shown in Table 9-2. Although passing the California WET was not a goal of this demonstration, additional processing of the soil (with the associated higher cost) may be required at other sites that are subject to more stringent regulatory requirements.

Table 9-1. List of Inorganic Persistent and Bioaccumulative Toxic Substances and Their Soluble Threshold Limit Concentration (STLC) and Total Threshold Limit Concentration (TTLC) Values

Substance	STLC (mg/L)	TTLC Wet Weight (mg/kg)
Antimony and/or antimony compounds	15	500
Arsenic and/or arsenic compounds	5.0	500
Asbestos	—	1.0 (as percent)
Barium and/or barium compounds (excluding barite)	100	10,000
Beryllium and/or beryllium compounds	0.75	75
Cadmium and/or cadmium compounds	1.0	100
Chromium(VI) compounds	5	500
Chromium and/or chromium(III) compounds	560	2,500
Cobalt and/or cobalt compounds	80	8,000
Copper and/or copper compounds	25	2,500
Fluoride salts	180	18,000
Lead and/or lead compounds	5.0	1,000
Mercury and/or mercury compounds	0.2	20
Molybdenum and/or molybdenum compounds	350	3,500
Nickel and/or nickel compounds	20	2,000
Selenium and/or selenium compounds	1.0	100
Silver and/or silver compounds	5	500
Thallium and/or thallium compounds	7.0	700
Vanadium and/or vanadium compounds	24	2,400
Zinc and/or zinc compounds	250	5,000

Table 9-2. Leachable Lead Concentrations

Sample No.	TCLP (mg/L)				California WET (STLC) (mg/L)			
	Lead	Copper	Zinc	Antimony	Lead	Copper	Zinc	Antimony
Nov. 22, processed soil	0.47	0.022	0.15	0.68	9.4	2.5	<1	5.1
Nov. 22, processed soil replicate	3.6	0.38	0.30	0.036	19	3.1	<1	2.1
California Limit	5.0	N/A	N/A	N/A	5.0	25	250	15

The following regulations may apply to small-arms range soil processing by physical separation/ acid leaching type technologies:

❑ NEPA

❑ RCRA

❑ EPA Military Munitions Rule (40 CRF Part 260) and the DoD Military Range Rule (Deliberative Draft of 32 CFR Part 339)

❑ EPCRA

❑ CWA

❑ CAAA

☐ OSHA

☐ State/Local Regulations (e.g., Title 22 California Code of Regulations).

9.1.1 National Environmental Policy Act (NEPA)

NEPA must always be considered when dealing with maintenance and remediation activities associated with small-arms ranges. A blanket statement cannot be made as to its potential impact on range maintenance/remediation activities. Instead, each site-specific application must be evaluated. However, it may be possible to fulfill NEPA requirements by applying a Categorical Exclusion (CATEX) with a Record of Environmental Consideration (REC), as described in Chapter 4 of Army Regulation (AR) 200-2, because of the limited scope of many range maintenance or remediation projects.

9.1.2 Resource Conservation and Recovery Act (RCRA)

RCRA, which regulates the classification, treatment, storage, and disposal of solid and hazardous waste, has the single greatest potential impact on this technology type. The driver is that the lead in the soil could satisfy the criterion of a RCRA characteristic hazardous waste. In order to add some clarity and common sense to this issue, both the U.S. EPA and the DoD have released draft procedural rulings. The U.S. EPA finalized their proposed "Military Munitions Rule" on February 12, 1997 (40 CFR Part 260). It provided clarity to the issue of hazardous waste identification and management for military munitions, including small-arms ranges. It addressed issues beyond the scope of range maintenance. It provided regulatory tools to allow the military services to largely manage their munitions cradle to grave without having to subscribe to the cumbersome RCRA hazardous waste management standards. Moreover, it recognized the military as being the subject matter experts in this general subject, and would recognize DoD precedence in such matters if and once the DoD issued their own proposed standards via a set of regulations.

The DoD responded to the EPA draft ruling on March 19, 1996, with their proposed "Military Range Rule." Both the final U.S. EPA and proposed DoD rules indicate that, if maintenance activities occur on site at an "active" small-arms range, the soil is not considered a RCRA hazardous waste. Concern still remains as to whether all states will recognize this. Personnel working with small-arms ranges need to carefully review these rulings and ensure that they understand the concepts of "on site," "active," "inactive," and "closed." To determine the applicability of RCRA regulations, the term *maintenance* is used in this report to describe activities that do not fall under the RCRA regulations. Those that do are referred to as *remediation*. However, RCRA hazardous waste probably will be generated during remediation activities, and such wastes will have to be managed following the full RCRA protocols. Examples of such wastes include used personal protective equipment (PPE), organic materials, and process water.

Recycling lead-bearing materials from a small-arms range maintenance or remediation project is a cost-effective and environmentally protective approach. The total cost of shipping and processing at a recycling center will be competitive with disposal at a RCRA-permitted TSD facility, but recycling has the following added advantages:

❑ The material returns to a beneficial reuse and does not become a waste.

❑ Potential future liability is eliminated because the recovered lead mixes with lead from other sources to form a commercial product.

❑ Recycling reduces a variety of regulatory requirements such as manifesting waste in accordance with RCRA requirements and reporting transfers in accordance with SARA Title III requirements.

The recycling operation will not result in a net profit because of both the low concentration of lead in the wastestreams and the cost of shipping. Bullets removed from the small-arms range soil by physical separation typically contain 30 to 60% lead. The precipitate that results from the treatment of the acid leaching solution typically contains 1 to 5% lead. A lead recycler will charge a tolling fee to process material with a lead content below about 95%. Costs to transport the recovered lead-bearing materials from the site to the recycling facility must also be paid.

Recycling the lead-bearing materials must be done in a careful and responsible manner. The materials must be compatible with the requirements of the recycling facility. Potential recyclers should be contacted during the early stages of project planning to determine the availability and capability of their facilities. Typically, facilities require a sample of the material to allow compatibility testing in their laboratory. During the Fort Polk demonstration, some of the materials could not be recycled as planned, even though the lead content was typical of and similar to that in materials that were successfully recycled.

9.1.3 Emergency Planning and Community Right-to-Know Act (EPCRA)

EPCRA was promulgated to establish emergency planning criteria and ensure that communities are informed about hazardous materials in their areas. The EPCRA requires facility owners who accumulate hazardous materials in excess of threshold planning quantities (TPQs) and to report such presence to local fire-fighting and emergency response agencies. If the separation and leaching technology uses hazardous materials (e.g., acids, etc.) in amounts that exceed these limits, it will require reporting interaction with the installation's Environmental Management staff.

9.1.4 Clean Water Act (CWA)

The CWA sets standards and requirements for pollutant discharge. The National Pollutant Discharge Elimination System (NPDES) (40 CFR Parts 122 and 125) requires permits for the discharge of pollutants from any point source into the waters of the United States. General pretreatment regulations are enforceable standards promulgated under 40 CFR Part 403 for discharge to a publicly owned treatment works (POTW) and could be applicable or relevant and appropriate requirements (ARARs) if surface or groundwater remediation results in discharge. The CWA also applies to this type of technology. The leaching or soil washing element eventually will generate contaminated wastewater. The processing plants employed in this demonstration used closed loops and make-up water, but the final water discharge had to be disposed of in a proper manner. Under most circumstances, such water may not be allowed into

a storm sewer system. Some installation wastewater treatment plants may be capable of handling such wastewater, but others may not. The wastewater handling issue needs to be dealt with in accordance with the local wastewater treatment plant's capability and NPDES permit requirements. An equally important consideration is the surface runoff that will be generated from the wet processing involved in this technology. Special care must be taken to implement both spill prevention, control, and countermeasures plans, as well as stormwater pollution prevention plans.

During this demonstration, the non-recycled wastewater was treated and tested to meet the acceptance criteria for the base wastewater treatment plant. If the wastewater met the criteria, it was discharged to the sewer leading to the wastewater treatment plant. If not, it was hauled away by a licensed hazardous waste disposal contractor. If the precipitation step of the leaching plant is carried out effectively, most of the wastewater can be discharged to the sewer.

9.1.5 Clean Air Act Amendment (CAAA) and Occupational Safety and Health Act (OSHA)

The CAAA was promulgated to establish standards and methods to reduce air pollution. Portions of the CAAA that may influence maintenance of remediation at small-arms ranges include:

❏ National Ambient Air Quality Standards (NAAQS) apply to total suspended particulate, sulfur dioxide, nitrogen dioxide, carbon monoxide, ozone, and lead concentrations in ambient air and are not applicable to individual emission sources. "Prevention of significant deterioration" (PSD) regulations may apply preconstruction guidelines and monitoring to statutory sources.

❏ New Source Performance Standards (NSPS) were developed for specific industrial categories to provide a ceiling for emissions from new sources.

❏ National Emission Standards for Hazardous Air Pollutants (NESHAPS) regulate asbestos, beryllium, mercury, vinyl chloride, coke oven emissions, benzene, radionuclides, and inorganic arsenic.

The CAAA has two major issues associated with this type of technology. The first is the potential for acid fumes in the ambient atmosphere, and the other is for the presence of lead dust above the allowable limit. Perimeter monitoring was employed during the demonstration to evaluate fume and dust potential for OSHA-type concerns. This is very important as the results impact the level of PPE that the workers have to wear. If PPE requirements go above Level D, work efficiency may decrease and project costs will increase. Lead exposure in construction is addressed in 29 CFR 1926.62.

9.2 REGULATORY ISSUES ADDRESSED AT FORT POLK

9.2.1 National Environmental Policy Act (NEPA)

The required NEPA documentation was completed by Fort Polk and the site support contractor. AREC with appropriate CATEXs was executed on April 3, 1996, to include coordination with the United States Forest Service. The procedures described in AR 200-1 and 200-2 were complied with.

9.2.2 Resource Conservation and Recovery Act (RCRA), the EPA Military Munitions Rule, and the DoD Military Range Rule

This project was executed in accordance with the new guidance contained in the EPA and DoD rules. The range hosting the demonstration was an active range, and the effort supported normal range maintenance. The soil being processed in the demonstration was recycled back to the range. Any RCRA hazardous waste that was generated was handled under RCRA protocols. However, the site was not considered a TSD facility. Although the demonstration involved the handling and processing of a hazardous material, no treatment of any hazardous wastes occurred here, nor was any such waste accumulated on this site above federal limits or time storage restrictions. The hazardous waste generated at this location included contaminated soils from the acetic acid process, jig concentrate, and metals-laden precipitate.

Because the range was an active range, and was adjacent to other active ranges on both sides, the actual recycling/processing demonstration occurred at another location approximately 2 miles from the range. A detailed health and safety plan was prepared for the demonstration by BDM, Inc., the site support contractor. An impervious bermed asphalt operations pad was constructed with its own 30,000-gallon holding pond to provide post-secondary containment. A 6-foot-high chain link fence with a barbed wire outrigger surrounding this area was also provided. Background soil samples were collected from the demonstration site to provide data on the contamination levels that existed prior to the demonstration.

Because the asphalt pad was too small to house the field sample preparation equipment, most of this equipment was set up in an adjacent unpaved area allotted to Battelle by the site support contractor. Extreme caution was exercised by field personnel to prevent spills or leaks during sample processing. After the demonstration, the Battelle field team collected several samples of soil from the sample preparation area soil. As shown in Appendix G, all samples had insignificant levels of total and TCLP lead. At future sites, however, it is recommended that sample preparation be conducted on the asphalt pad itself because of the potential to contaminate the area while using the sample processing equipment.

9.2.3 Emergency Planning and Community Right-to-Know Act (EPCRA)

EPCRA involves the requirement for reporting hazardous materials that exceed TPQs. Provisions to do so were established by the site support contractor, and the Fort Polk Environmental Management staff were informed.

9.2.4 Clean Water Act (CWA)

With regard to CWA issues, both a site Solid Waste Pollution Prevention (SWPP) plan and a Spill Prevention, Control, and Countermeasures (SPCC) plan were prepared by the site support contractor and were in effect during the demonstration. The vendors' demonstration equipment had its own secondary containment. The containment pond at the end of the pad provided backup containment and served as a significant safety factor. This pond was piped to the sanitary sewer on post, but a valve was installed on the line, within the fenced secure area, to prevent accidental release. The plans called for treating this water to meet local POTW requirements. In some cases, site officials may allow accumulated rainwater to be discharged to their sanitary sewer, if the water is not so contaminated as to impede wastewater treatment. The Fort Polk wastewater treatment plant has a trickling filter system and is NPDES permitted.

9.2.5 Clean Air Act Amendment (CAAA) and Occupational Safety and Health Act (OSHA)

Air sampling and monitoring were conducted at the site. The target pollutants of concern were lead dust and acid fumes. The provisions of 29 CFR 1926.62 (lead in construction) were being implemented via the Health and Safety Plan (HASP). Level D PPE was the norm, although the potential existed for the site workforce to wear Level C PPE.

9.3 COMMUNICATING WITH THE PUBLIC AND OTHER DoD SITES

The installation commander, through his Environmental Management staff, was the authorized line of communication to all environmental regulatory offices on matters that pertained to this installation. A letter fully describing the scope of this demonstration project was submitted. The installation had an active Environmental Quality Control Committee (EQCC) that met monthly. In addition, the installation held Remediation Advisory Board (RAB) meetings during which the local community and regulatory officials participated.

Another user participation event was the Visitors Day coordinated by Battelle and held on December 12, 1996. During this event, potential users, along with the regulatory community, were invited to visit the site and were given briefings and guided tours. In addition, informational project brochures were developed and used as handouts.

10. Application at Future Sites and Technology Transfer

10.1 CHARACTERISTICS OF DoD SMALL-ARMS RANGES

The demonstration of the physical separation and acid leaching technology at Fort Polk was conducted to evaluate a potentially cost-effective option with potential for cleaning up the more than 2,600 small-arms ranges operated by DoD. Small-arms ranges in use by the DoD today have been in operation for many years without a comprehensive policy on preventive range maintenance to address lead and other metal contaminants. Because of the inevitable buildup of bullets in impact berms, these ranges are potential sources of metals contamination. If left unattended, the metals potentially may be transported into the environment along various pathways including surface water runoff, groundwater migration, and airborne dust migration.

Outdoor small-arms ranges are designed to allow the firing of bullets of 50 caliber or less (e.g., pistols, rifles, shotguns, submachine guns, and machine guns) for a variety of training and weapons qualification missions. The types of ranges that are encountered at DoD facilities include but are not limited to the following:

☐ Long-distance ranges for high-powered rifles
☐ Short-distance ranges for pistols and battle sight zero (BZO) operations
☐ Ranges for combat training and simulation
☐ Trap and skeet ranges for shotguns.

Details on range configuration are provided in a Military Handbook (DoD, 1992). Despite the variety of configurations, all conventional ranges have an impact berm or a projectile fall area where bullet metals accumulate. At most ranges, the berm or fall area consists of soil. In a few cases, the bullet collection area is a body of water.

An NFESC survey of Navy and Marine small-arms activities (NCEL, 1991) documented the characteristics, configurations, and amount of contaminated soil at small-arms ranges. A questionnaire requesting information about the configuration of small-arms ranges was sent to 65 sites to provide an initial basis for estimating the typical size and configuration for berms. Berms were found in many sizes, with heights varying from as low as 5 ft to as high as 50 ft and lengths varying from 15 ft to 5,280 ft. The mean height, width at the base, and length of the berms reported are 18 ft, 42 ft, and 340 ft, respectively. However, this sample set was not very normally distributed. A large number of the sites had smaller berms and a few sites had a large berms, thus giving a bimodal appearance to the size data distributions.

Table 10-1 gives a summary of the berm dimensions data collected in the survey. The median values are tabulated along with the 10th and 90th percentile to better describe the distribution. Because of the large spread of the data, the median is probably a better statistic than the mean. The median berm volume is 2,000 yd^3 (2,700 tons), which is about twice the volume of soil processed during the hydrochloric acid demonstration.

Table 10-1. Summary of Small-Arms Range Dimensions Data

Parameter	10th Percentile	Median	Mean	90th Percentile
Number of firing points	<6	11	18	>45
Height (ft)	<8	15	18	>30
Width (ft)	<10	25	42	>90
Length (ft)	<50	100	340[a]	>450
Total volume (yd³)	<200	2,000	17,500[a]	>14,000

(a) Includes berms with lengths of 5,280 ft and 1,500 ft. The average length and volume without these two berms are 130 ft and 3,100 yd³, respectively.

10.2 PHYSICAL SEPARATION/ACID LEACHING FEASIBILITY AT OTHER DoD SITES

The physical separation and acid leaching technology is potentially applicable for maintenance or remediation at many of these sites. The main contaminants of interest at a small-arms range remediation are lead, copper, zinc, antimony, and arsenic in soil or sediment. Physical separation followed by acid leaching is the only well-demonstrated technology that can remove the metal contaminants from the soil for recycling. The processed soil is physically and chemically suited for return to an active berm. The variety of beneficial uses that the site can be put to is greater because the lead and other contaminants have been mostly removed.

The innovative physical separation and acid leaching technology is less familiar to Remediation Program Managers (RPMs) than conventional approaches such as S/S treatment or excavation and disposal. This physical separation/acid leaching demonstration aimed to collect and document cost and performance data to improve RPM acceptance of the technology and promote widespread implementation. Section 10.3 describes some of the technology transfer efforts planned to transition this technology to potential users.

The different types of soil and lead species at other DoD sites may not be a significant limitation for this technology if a strong acid (such as hydrochloric) is used. However, if the use of a weak acid (such as acetic) is desired, there may be a limitation based on economics (because of high acid volume requirement) and based on the lead species and the way they are bound to the soil matrix. However, even hydrochloric acid may be challenged if lead is present in particularly recalcitrant forms, such as lead sulfate. Section 9.1 discusses some of the regulatory issues that may affect the technical feasibility and cost of physical separation/acid leaching at sites located in certain states of the United States, where cleanup targets are more demanding.

Cost is another important issue governing implementation of the technology. As seen in Table 10-1, many sites have smaller berms. From a cost standpoint, at smaller inactive ranges, it may appear attractive to haul the soil to an off-site landfill. At many active ranges, periodic dry screening of the berm soil to remove bullets (at less than $50/ton) may provide adequate cleanup

for preventive maintenance. If additional metal mobility reduction is required, S/S treatment may be a slightly less expensive short-term option than physical separation/acid leaching. However, at most medium- and large-sized ranges (over 1,000 or 1,500 tons), the physical separation and acid leaching technology is cost-competitive with both landfilling and stabilization. If a true cost-benefit analysis is done, even smaller sites may find physical separation/acid leaching to be a more beneficial solution that is more environmentally protective and permanent.

10.3 TECHNOLOGY TRANSFER/IMPLEMENTATION

10.3.1 Status of Acetic Acid Process Vs. Hydrochloric Acid Process

Based on the results of the Fort Polk demonstration, further pursuit of an acetic acid process will require additional bench and pilot demonstrations prior to implementation. However, the hydrochloric acid process is ready for implementation and is not expected to require further development or demonstration.

10.3.2 Technology Transfer Vehicles

Because the technological know-how of the hydrochloric acid process is available commercially, what is needed is a method to transfer the Fort Polk experience and lessons learned to various vendors and potential users to assist them in implementing this knowledge at other small-arms ranges. This can best be accomplished through wide distribution of the Fort Polk reports, which include this Technology Evaluation Report, the Implementation Guidance Handbook, and the Technical Application Analysis. In addition, other forums for communicating this experience, such as tiger teams and technology seminars, may be used.

To facilitate the technology transfer process, Battelle organized the following items:

☐ Visitors Day on December 12, 1996 during the Fort Polk demonstration: Battelle coordinated a Visitors Day at Fort Polk while Vendor 2 was processing at the site. Several hundred invitations were sent to potential DoD users, site managers, regulators, and other interested individuals. Presentations and guided tours were organized at the site to give visitors a fair understanding of the technology and its implementation.

☐ Introductory Brochure on the Fort Polk Demonstration: Battelle prepared a four-page color brochure describing the activities under way at Fort Polk for demonstrating the physical separation/acid leaching technology for small-arms range maintenance. This brochure had detailed descriptions of the two vendors' plants to introduce potential users and vendors to the technology and its application. This brochure was distributed to the attendees of Visitors Day, as well as to several other potential users.

☐ Final Brochure on the Fort Polk Demonstration: Battelle prepared a fold-out color brochure to summarize the technology and the demonstration results. This brochure will be distributed to DoD site managers, regulators, and other interested parties.

☐ Initial Video Film: An informational video film showing demonstration activities was prepared by Battelle at the end of the demonstration. This video film, will be used at technology transfer and training seminars.

☐ Final Video Film: A second video film summarizing the activities at the Fort Polk demonstration is being prepared by Battelle. This film also will summarize the results of the demonstration, and will be used in technology transfer and training seminars.

10.3.3 Guidance for Vendor Selection and Evaluation

To implement this technology, it is recommended that suitable vendors be used rather than have DoD or the sites purchase and operate a processing plant. Several vendors were identified in either the mining or the remediation industry. Remediation vendors have been using mining processes for soil washing-type applications for many years now. However, the vendor evaluation and selection process is critical. At Fort Polk, the success of the hydrochloric acid process and the difficulties encountered in the acetic acid process can be at least partially attributed to the individual vendor's capabilities.

During the vendor selection process the following issues should be taken into consideration:

☐ The vendor must have experience with physical separation and acid leaching processes for metals-contaminated soils (experience with lead is preferred).

☐ Performance specifications rather than equipment specifications should be used to take advantage of the vendor's experience.

☐ The vendor must have an on-site supervisor who has experience in plant design and operation so that troubleshooting and modifications can be conducted if required.

☐ The vendor must have a chemist at the site for process control. An on-site AA unit would be desirable.

☐ Before field operations begin, the vendor must provide a process flow sheet with material balance information. An oversized plant is necessary to handle variations in feed soil texture and contaminants.

☐ The vendor must ensure that potential material handling difficulties have been identified and addressed.

Selected vendor(s) should be asked to perform meaningful bench-scale tests taking the following issues into consideration:

☐ The site should ensure that the vendor receive representative samples of the berm soil. A composite sample of approximately 30 gallons collected from grab samples taken from a number of locations in the berm is desirable. A separate sample from the most contaminated regions of the berm should also be tested.

❏ The vendor should perform testing of all critical unit operations planned at the site; including:

- Screening (dry or wet)
- Other physical separation (e.g., density, hydrodynamic, magnetic, froth flotation) as required
- Acid leaching (e.g., pH, contact time, number of contacts)
- Regeneration of the acid leaching solution (e.g., precipitation, solid-liquid separation)
- Dewatering and neutralization of the processed soil.

11. Conclusions and Recommendations

Based on the results of the Fort Polk demonstration, the physical separation and acid leaching technology can be said to be an attractive option for small-arms range maintenance or remediation operations. The technology has the following benefits:

☐ Significant amounts of lead, as well as other heavy metals of concern, are removed by the process from the range soils. Most of the lead removed can be successfully recycled in an off-site smelter. This makes physical separation/acid leaching a long-term remedy with a low potential for future liability. The physical form and chemical composition of the processed soil make it suitable for reuse in an active range. Treatments of inactive ranges with this technology will allow the reuse of the site for a greater variety of purposes than would be possible with other on-site treatments, such as S/S.

☐ The technology is competitive in cost with other commercial options, such as on-site stabilization or off-site landfilling. At sites with less than about 5,000 tons of contaminated soil, off-site landfilling costs less in the short term because an off-site remedy incurs lower fixed (capital) costs. However, the long-term benefits of removing and recycling the lead may still make physical separation/acid leaching an attractive option for ranges overall.

☐ Implementation of this technology is facilitated by the fact that there are vendors in the United States who are in a position to quickly assemble a commercial plant capable of conducting the processes associated with physical separation/acid leaching. Regulatory acceptance of this technology, which has considerable short-term and long-term benefits, should also ease the implementation process.

11.1 SUMMARY OF CONCLUSIONS FROM THE FORT POLK DEMONSTRATION

Two vendors demonstrated their respective versions of the physical separation and acid leaching technology. Vendor 1 used weak (acetic) acid leaching and Vendor 2 used strong (hydrochloric) acid leaching. Based on the results of the evaluation conducted by Battelle, several conclusions can be made regarding the demonstration and are contained in the following sections.

11.1.1 Site Preparation and Support

The mission support and program integration contractor at Fort Polk conducted the following activities to support the demonstration of separation/leaching for Range 5 maintenance:

☐ Prepared the required regulatory documentation, including an environmental assessment, and obtained the required approvals.

☐ Conducted a worldwide search that identified and taxonomically categorized the state of this technology set in the commercial marketplace.

☐ Constructed an asphalt pad with appropriate containment to house the vendors' plants and arranged for the utilities and disposal of residuals from the first process. A trailer and covered shelter were provided to house offices and the small on-site analytical laboratory.

☐ Arranged for the excavation, delivery, and return of the berm soil used during processing.

These support activities greatly facilitated the logistics of the plants' operations and the controlled disposal of residuals. Minor improvements that could be suggested include:

☐ The need for additional soil storage space on the pad to account for the turnaround time requirement for TCLP verification.

☐ Relocation of some supply elements, such as the acid storage tank, on the pad. The locations used for some of these elements during the demonstration necessitated the passage of chemical supply trucks through the exclusion zone.

☐ Ensuring adequate power supply; the power supply provided at the site did not adequately support all the requirements of the vendors' plant and the on-site analytical equipment. Some excess capacity is desirable for changes or additions to the plant that may be made if the initial process configuration runs into difficulties. At Fort Polk, diesel generators were rented by the vendors and Battelle to supply additional power needs.

In general, on-site support personnel ensured smooth implementation of the demonstration activities. An on-site health and safety officer arranged for ambient air monitoring for lead to ensure worker safety during the demonstration.

11.1.2 Vendor 1 and the Acetic Acid Process

Figures 11-1 and 11-2 summarize the results of the acetic acid process demonstrated by Vendor 1. On the first day of processing, the processed soil met both total and TCLP lead criteria, indicating that the acetic acid leachant can be effective with small-arms range soils. However, on subsequent processing days, the plant was unable to meet the TCLP criterion.

Vendor 1's demonstration was hampered by several process design and plant operation difficulties. The difficulties may have been partly the result of an aggressive schedule and the fact that Vendor 1 was assigned to perform the first demonstration. However, many of the problems can be traced to the vendor's bench-scale study and the process control and material handling problems encountered in the field.

(a) Field Demonstration Total Metals Pb Concentrations for Processed and Raw Soil

(b) Field Demonstration Total Metals Cu Concentrations for Processed and Raw Soil

(c) Field Demonstration Total Metals Sb Concentrations for Processed and Raw Soil

(d) Field Demonstration Total Metals Zn Concentrations for Processed and Raw Soil
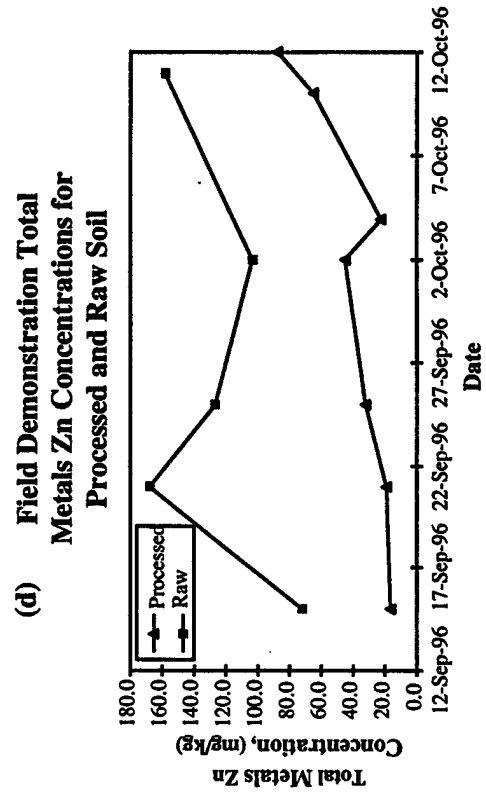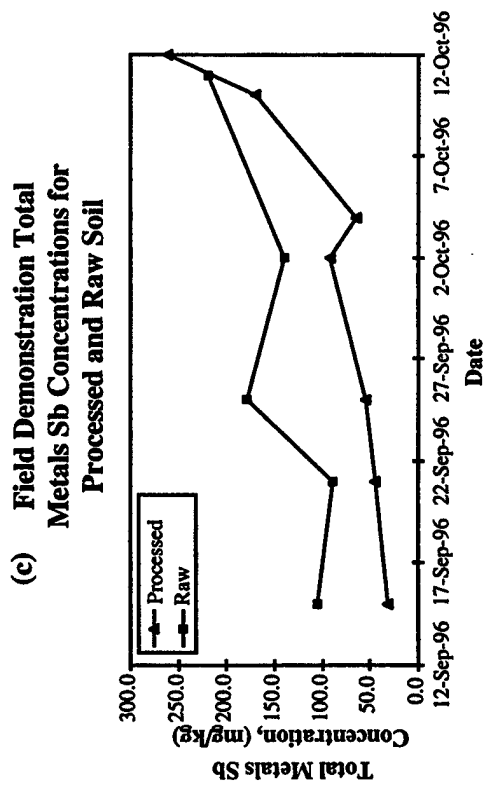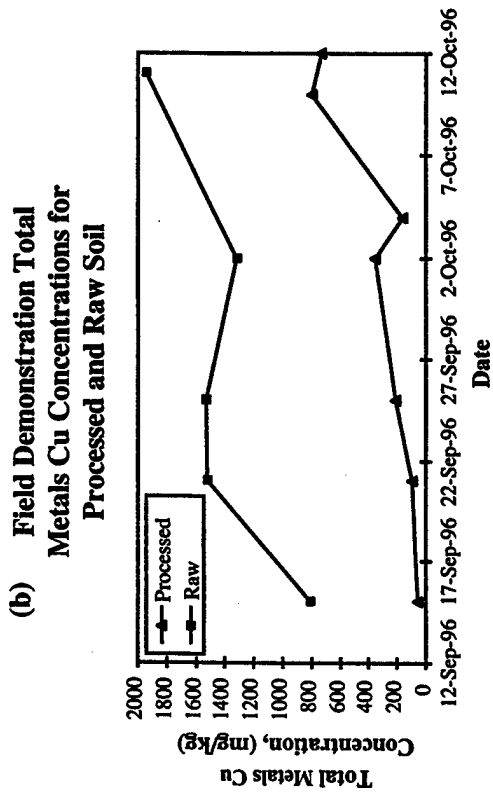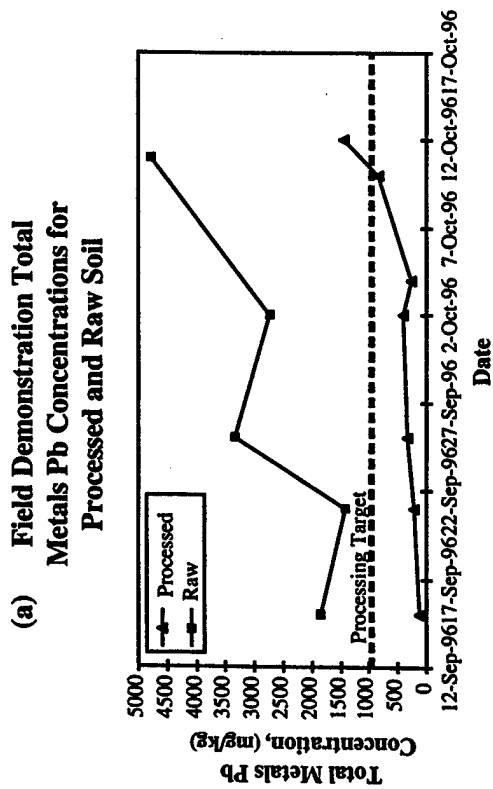
Figure 11-1. Total Metals Removal During the Acetic Acid Demonstration at Fort Polk

(a) Acetic Acid Field Demonstration
TCLP Pb Concentrations for
Processed and Raw Soil

(b) Acetic Acid Field Demonstration
TCLP Cu Concentrations for
Processed and Raw Soil

(c) Acetic Acid Field Demonstration
TCLP Sb Concentrations for
Processed and Raw Soil

(d) Acetic Acid Field Demonstration
TCLP Zn Concentrations for
Processed and Raw Soil

Figure 11-2 TCLP Performance During the Acetic Acid Demonstration at Fort Polk

☐ The bench-scale testing did not adequately address the precipitation element. Inadequate precipitation of lead resulted in the persistence of high levels of dissolved heavy metals in the regenerated leachant. This was the biggest contributor to the inability of the processed soil to meet the TCLP target. The proprietary precipitant used has a pH optimum of 8.5. The bench-scale testing did not adequately investigate the operating pH range, precipitant dosage, flocculation, and dewatering associated with precipitation.

☐ Inadequate process control during the field operation prevented the cause of the operating difficulties from being properly identified. A full-time on-site process chemist using the AA unit would have helped identify the high levels of lead in the regenerated leachant.

☐ Material handling problems were encountered in several process elements, including the feed hopper and the plate feeder and press. In addition, other pieces of equipment seemed to be undersized, which caused bottlenecks in the process that slowed or shut down the operation. The plant required 14 days for mobilization, following which it was operational 65% of the time. The plant managed an average feed rate of 3.0 tons/hr, although it is unlikely that the continuous plant reached a steady-state operation during the demonstration.

☐ Inadequate dewatering/neutralization of the processed soil occasionally created hazardous working conditions due to the excess acid vapors. Sampling personnel had to upgrade to Level C PPE to approach the processed soil pile for sample collection.

11.1.3 Vendor 2 and the Hydrochloric Acid Process

Vendor 2 had some procedural advantages over Vendor 1 because of longer preparation time, advance knowledge of the problems encountered by Vendor 1, and the use of the stronger acid. However, Vendor 2 also performed a more comprehensive bench-scale study that evaluated all elements of the process. During the field operation, a process chemist with an on-site AA and XRF unit ensured that operating conditions determined during the bench-scale testing were met. The plant was mobilized in 18 days, after which it was operational for 94% of the time.

Figures 11-3 and 11-4 summarize the results of Vendor 2's demonstration. Total and TCLP lead targets were consistently met and the plant was easily able to handle the feed soil and contaminant variability. The processed soil was dewatered, neutralized, and returned to the active berm. The recovered metals and precipitate were sent to an off-site smelter for recycling and reuse of the lead content, and 22,000 gallons of process solution having a lead concentration of less than 5 mg/L was discharged to the POTW at demobilization. The organic matter collected during operations was reblended with the processed soil and returned to the range. However, at future sites, this organic matter may be better disposed of with the precipitate sludge because of its high total and TCLP lead levels.

The total cost of implementing hydrochloric acid processing for routine maintenance with Vendor 2's plant at a site containing 835 tons of contaminated soil (the amount processed at Fort Polk) is estimated to be $1.2M or $1,400/ton. This assumes that the soil is processed to meet the TCLP target of 5 mg/L lead. At a larger site, having 10,000 tons of contaminated range soil, the

(a) Field Demonstration Total Metals Pb Concentrations for Processed and Raw Soil

(b) Field Demonstration Total Metals Cu Concentration for Processed and Raw Soil

(c) Field Demonstration Total Metals Sb Concentrations for Processed and Raw Soil

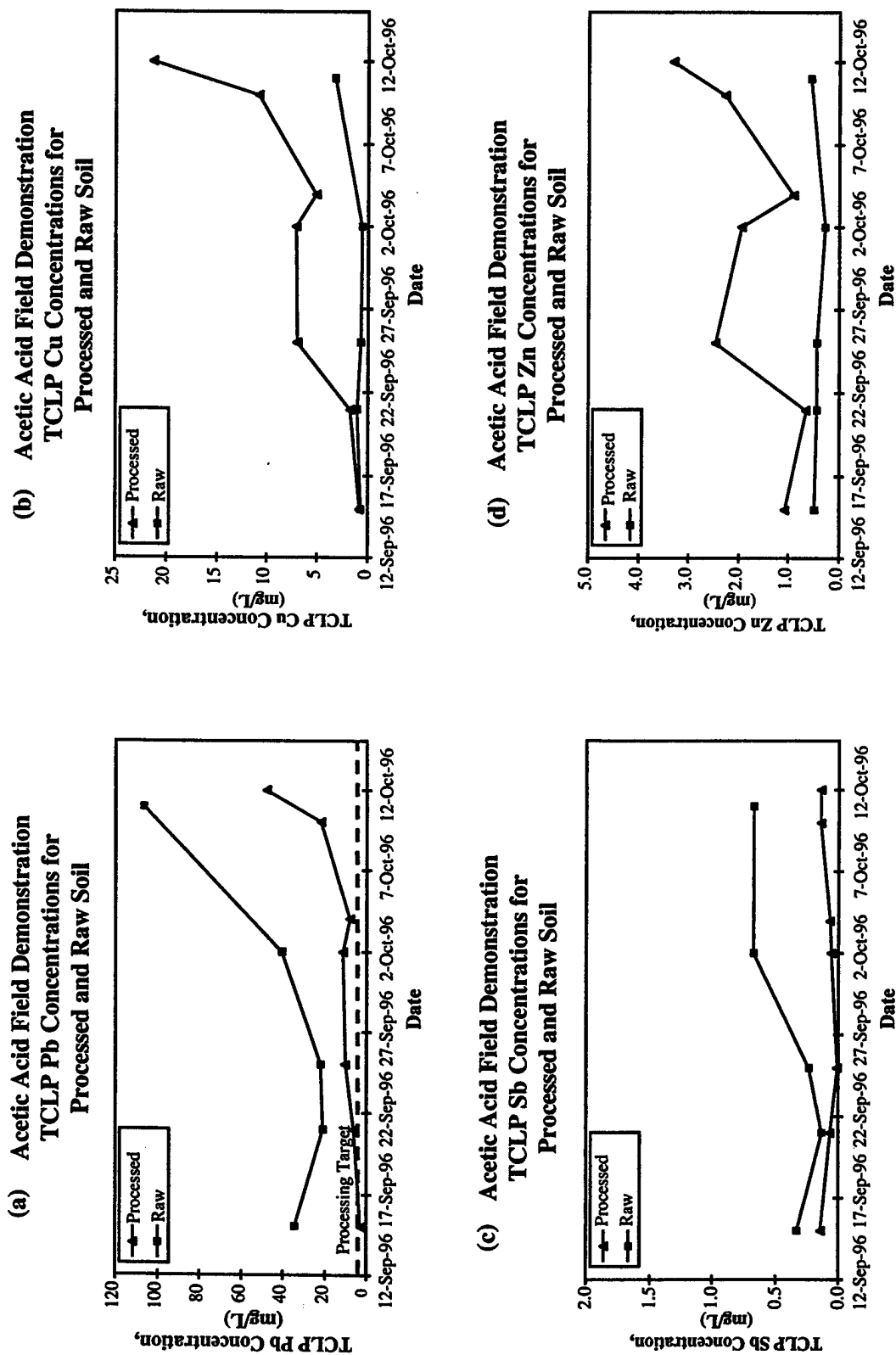**(d) Field Demonstration Total Metals Zn Concentrations for Processed and Raw Soil**
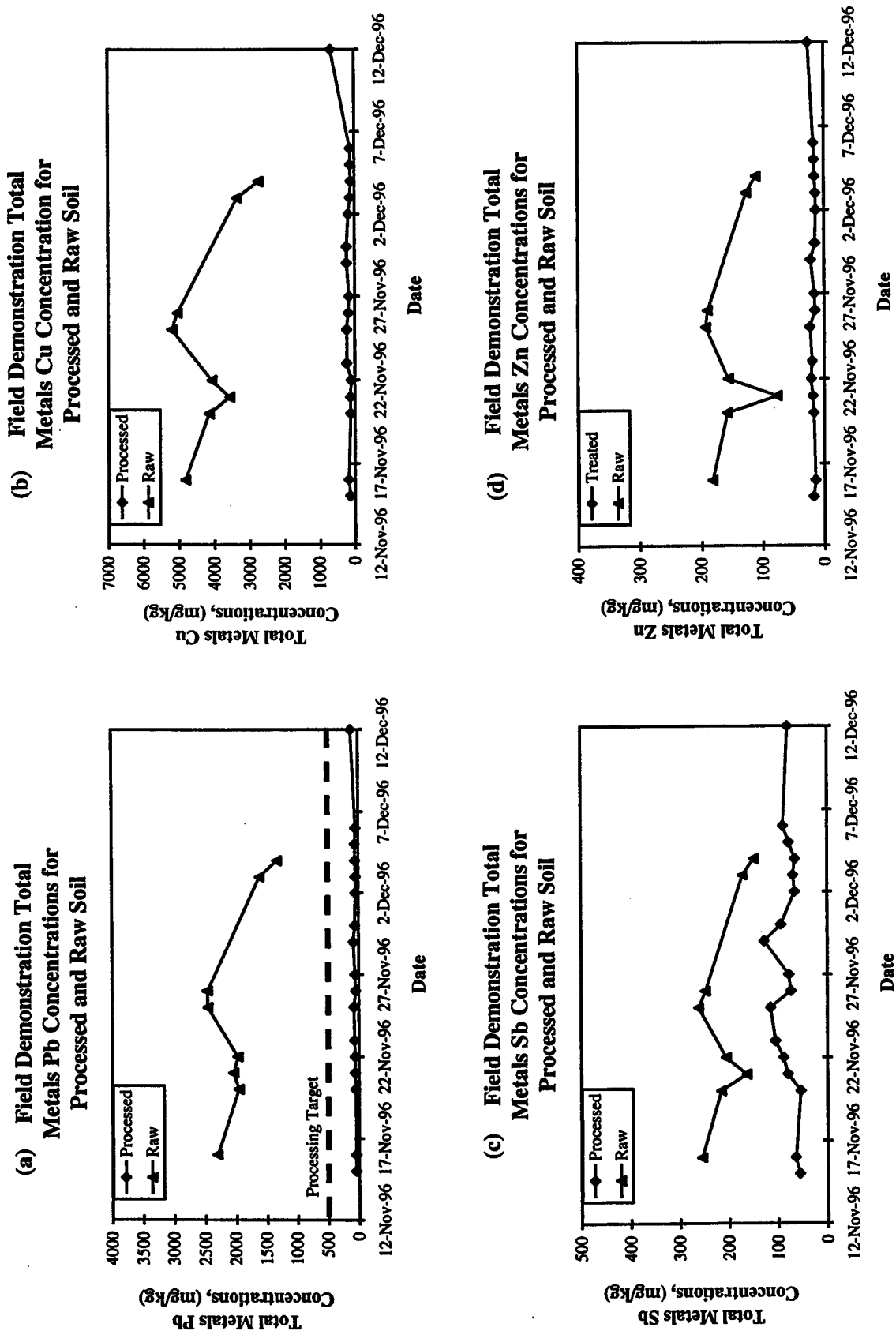
Figure 11-3. Total Metals Removed During the Hydrochloric Acid Demonstration at Fort Polk

BATTELLE

(a) Hydrochloric Acid Field Demonstration TCLP Pb Concentrations for Processed and Raw Soil
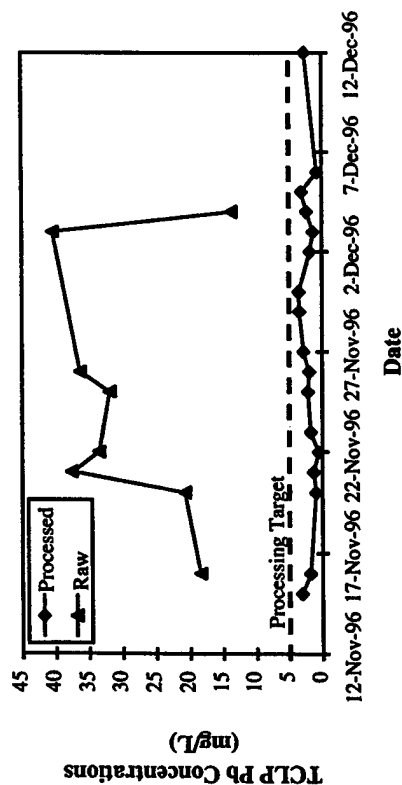
(b) Hydrochloric Acid Field Demonstration TCLP Cu Concentrations for Processed and Raw Soil
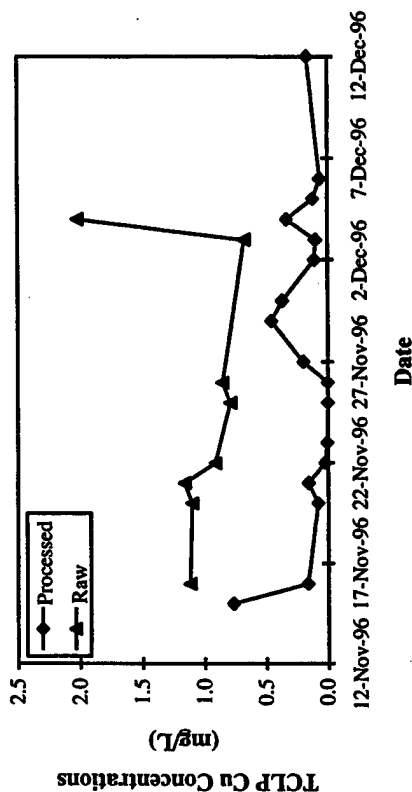
(c) Hydrochloric Acid Field Demonstration TCLP Sb Concentrations for Processed and Raw Soil
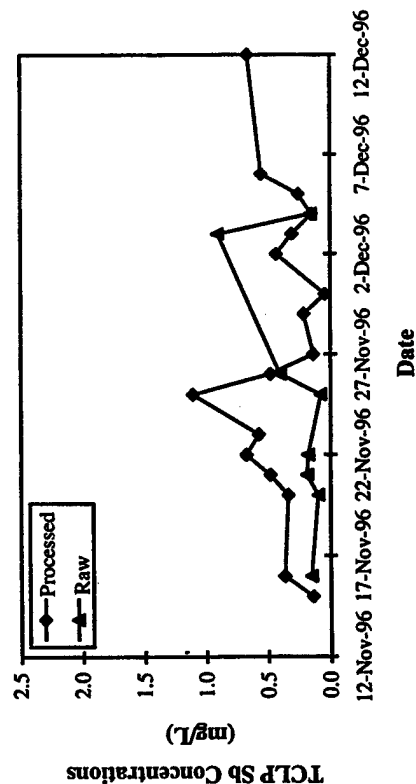
(d) Hydrochloric Acid Field Demonstration TCLP Zn Concentrations for Processed and Raw Soil

Figure 11-4. TCLP Performance During the Hydrochloric Acid Demonstration at Fort Polk

projected cost is $1.61M at $169/ton. The high fixed cost of implementing an on-site remedy makes the process more cost effective as the amount of material processed increases.

11.1.4 Acetic Acid and Hydrochloric Acid as Leachants

In general, the amount of acetic acid required to reach and maintain the pH (3.2 to 3.4) required for efficient leaching was high; large quantities of this acid were used during the demonstration. Depending on the types of soil and lead species at 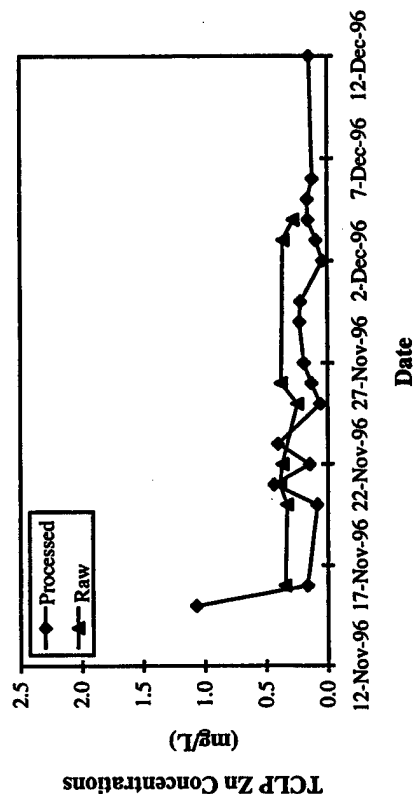other sites, even lower pH may be required. Because of the operational difficulties encountered by Vendor 1, a good estimate of acetic acid processing costs could not be obtained. Given the higher unit price of acetic acid, and the similarity of the two vendors' plants, preliminary indications are that acetic acid processing will be more expensive than hydrochloric acid processing.

Acetic acid, being a weak acid, dissociates only partially. Large quantities of acetic acid are required to attain the low pHs required for efficient leaching as shown in Table 11-1. Acetic acid is, however, more benign on plant equipment and soil structure than hydrochloric acid. The use of hydrochloric acid could shorten the useful life of process equipment. Alternatively, the hydrochloric acid plant may have to use more expensive stainless steel equipment.

Table 11-1. Strengths of Various Molar Concentrations of Acetic and Hydrochloric Acid

HCl Solution	pH	Acetic Acid Solution	pH
0.100M	1.00	0.100M	2.87
0.0100M	2.00	0.0100M	3.37
0.00100M	3.00	0.00100M	3.90

11.2 RECOMMENDATIONS FOR USE OF THE SEPARATION/ LEACHING TECHNOLOGY AT SMALL-ARMS RANGES

The Fort Polk demonstration provided a good basis for projecting the requirements and considerations for implementing physical separation/acid leaching at future small-arms range sites.

11.2.1 Regulatory Setting

At active ranges, soil processing activities can be implemented as range *maintenance* under the EPA Military Munitions Rule and the DoD Military Range Rule, as long as the processed soil is reusable in the berm. At inactive ranges, any soil processing is likely to come under RCRA, with all its associated requirements. Range *remediation* under RCRA would require that the process soil meet the TCLP limit for lead (less than 5 mg/L). In addition, states such as California, may have more stringent requirements for the processed soil to meet. In California, copper, zinc, and antimony are environmentally regulated metals. The California WET, which uses stronger

leaching conditions than the TCLP test, is used to determine acceptable levels of heavy metals in remediated soil.

It is expected that although range maintenance may not come under RCRA or state regulations, a good faith attempt will be made to ensure that the processed soil meets at least the TCLP lead level of 5 mg/L. The processing conducted at Fort Polk, and all associated cost projections, are based on this assumption. At inactive ranges, if regulatory targets for on-site reuse of the processed soil are significantly lower than about 150 mg/kg total lead and 5 mg/L (TCLP) leachable lead, the technical and cost challenges facing the technology will increase. Therefore, it is important to conduct a comprehensive bench-scale treatability test on a site-specific basis to ensure that the required processing targets are achievable and that the associated costs appear reasonable. As a last resort, the final processed soil after utilizing physical separation/acid leaching technology may be stabilized with an environmentally compatible reagent, such as phosphate, in order to meet more stringent regulatory requirements. At most sites, however, the physical separation/ acid leaching technology is likely to provide a technically feasible and cost-effective option that is attractive to both site managers and regulators.

11.2.2 Characterization of Range Soils and Contaminants

Range soils are often very heterogeneous in texture. During, its history, a small-arms range berm may have received several layers of soil from different sources, some of which may not even be native soils from the site. In the past, range maintenance has sometimes included screening of the bullet pockets and adding a fresh layer of soil. Soil texture, or particle size distribution, is important for the contracted vendors to know in order to design an effective processing plant. The processing rates of several plant elements, such as screening or leaching, are dependent on the particle sizes encountered. For example, leaching rates are slower for finer soil fractions because of the stronger tendency of fine soil to sequester heavy metals.

Because of the heterogeneous distribution of soil and contaminants in the prospective berm, several grab samples from different parts of the outlined excavation pattern should be collected and composited. The size of the composite is determined by the maximum particle size of lead fragments expected. Based on the experience at Fort Polk, a 30-gallon (approximately 300 lb) drum filled with composited soil gives a good representation of raw berm soil. This composite sample can be analyzed by the site or by the vendor. The vendor will require such a composite sample to conduct bench-scale tests and design the processing plant. Alternatively, several grab samples from different locations in the berm may be collected and analyzed separately.

Wet screening provides a more reliable particle size analysis than dry screening. At Fort Polk, dry screening resulted in a tendency to underestimate the proportion of fines (–200 mesh) in the berm soil. The reason for this is that in dry sieving, balls of agglomerated clay stay on the coarse screens as oversize. Finer soil not only requires higher residence time in many processing elements, it can also create material handing problems of the type encountered during Vendor 1's processing.

11.2.3 Vendor Contracting

Several vendors in the mining and remediation industries appear to have the type of equipment and capabilities required for this technology. Sites can therefore contract the services of such vendors, rather than build and operate their own plants. Based on the Fort Polk experience, the vendor selection and contracting process should address the following elements:

☐ Vendors should have prior mining or remediation experience with physical separation/acid leaching type technologies. A solicitation of interest to prospective vendors may include preliminary site characterization data, an estimate of the amount of contaminated material that requires processing, and the maximum number of days an active range can be shut down for maintenance. Vendors would respond with a preliminary capabilities package and a budget estimate.

☐ Candidate vendor(s) should be given a representative sample of the range soils for characterization and bench-scale testing. A comprehensive bench-scale evaluation that simulates all elements of the proposed process should be conducted. A bench-scale testing report that outlines the type of processes, reagents, and operating conditions should be submitted. This should ensure that the vendor has evaluated all aspects of the process before mobilizing to the site to conduct operations.

☐ The selected vendor should be asked to provide a complete process flow diagram indicating all input, output, and intermediate streams. Supporting information should include estimated material flow and particle size composition for each stream. The design should include excess capacity to handle the variability in the feed soil that could occur.

☐ The plant design should include information on site preparation needs and utilities support required from the operations site.

11.2.4 Bench-Scale Testing and Plant Design

Bench-scale testing on a site-specific basis is essential for this technology. The success of the hydrochloric acid demonstration and the deficiencies of the acetic acid demonstration at Fort Polk can be traced partly to the respective vendors' bench-scale tests. Vendor 1 did not do a comprehensive evaluation of the precipitation element. In the field, inadequate precipitation resulted in the persistence of heavy metals in the regenerated leachant. This led to a general decline in the process performance over time.

Most bench-scale tests should start with a characterization of a representative sample of the range soil as described above. In addition to the particle size analysis and total and TCLP metals analysis of the composite sample, the concentration of total and TCLP metals in the different size fractions (gravel, coarse sand, fine sand, and fines) should be determined. Each soil fraction can also be tested by TCLP. Processing can then be focused on fractions that fail the TCLP test. All elements of the proposed plant should be evaluated, including:

- Feed control
- Size separation
- Gravity separation
- Leaching (choice of leachant and operating conditions)
- Precipitation and regeneration of leachant
- Dewatering
- Other (e.g., neutralization or stabilization of the processed soil if required).

Whether or not additional characterization of the range soil is required will depend on the results of initial bench-scale tests. Vendor 2, who used a stronger (hydrochloric acid) leachant, had no difficulty in getting the soil to pass the TCLP test during bench-scale testing. Beyond conducting particle size analysis of the raw soil and total and TCLP lead analyses of each size fraction, Vendor 2 did not have to scrutinize the raw material any further to reach processing targets. However, Vendor 1, who used a weaker (acetic acid) leachant, had more difficulty meeting the TCLP target. Vendor 1 could have benefited from a more detailed characterization of the raw soil, and so probably would sites that decide to use a weak acid leachant. Some sites may prefer a weak acid leachant because strong acids, such as hydrochloric acid, are more aggressive on both soil structure and processing equipment.

Additional soil and contaminant characteristics that may help optimize leaching effectiveness are:

- Cation Exchange Capacity (CEC) of Soil. CEC is an inexpensive parameter routinely measured by several commercial laboratories. It indicates the potential of the soil to bind heavy metals in an exchangeable form.

- Total Organic Carbon (TOC). TOC is a routine inexpensive parameter to measure. It will give the organic matter content in the soil. Organically complexed lead is harder to remove from the soil, especially with a weak acid.

- Iron and Manganese Content. This is an inexpensive, routine analysis to perform and will indicate potential presence of hydrous ferric oxide (FeOOH) or manganese oxide (MnO_2), both of which have the potential to strongly adsorb heavy metals.

- Heavy Metal Speciation. This is a complex and relatively expensive characterization that requires specialized methods, such as XRD or SEM. These methods provide an understanding of the chemical species of the heavy metals in the soil. Some lead species, such as carbonates, are more readily leached than other species, such as oxides or sulfates. Recently, researchers at DuPont and Hazen (Igwe et al., 1994) have developed a sequential extraction procedure for lead-contaminated soil that identifies the different ways in which lead is bound to the soil matrix. This procedure may help select appropriate leachants.

Bench-scale testing and plant design are reciprocal elements. Bench-scale testing is conducted to simulate a preliminary conceptual design of the various elements of a field-scale plant. The field

plant is then designed based on bench-scale verification of the conceptual design. The plant design should take the following additional issues into account:

☐ Plant Size. The plant should be oversized to accommodate inevitable variability in the feed soil texture and contaminant loading. Several pieces of Vendor 1's plant seemed undersized and resulted in bottlenecks at intermediate points that slowed down or halted the processing. Higher levels of fines or higher heavy metal content in the feed, or intermediate streams may require additional residence time in key process components, such as the leaching tanks.

☐ Material Handling. Material handling difficulties were encountered in Vendor 1's plant at several points, including in the feed hopper, plate feeder, attrition scrubber, and screen deck and filter presses. Soils with high fines content generally tend to be more difficult to handle, especially in the wet processes associated with this technology, because fines can agglomerate, stick to equipment, and resist solid-liquid contact and dewatering. Material handling problems can hamper process efficiency and increase downtime; therefore, they need to be addressed early in the bench-scale study and plant design.

11.2.5 Site Preparation

The site or site support contractor will have to provide the required base support for the vendor's operation. This may include:

☐ Addressing any regulatory issues, such as environmental assessments, discharge permits, etc.
☐ Building an asphalt pad to house the process plant
☐ Building appropriate containment around the process plant
☐ Providing power and water supply to the plant and other associated site needs
☐ Arranging for ambient air monitoring during operations
☐ Arranging for the disposal of process residuals generated during operations.

Some of these activities, such as ambient air monitoring and disposal of residuals, may be included in the vendor's contract, if desired. Excess capacity in the utilities arranged for would be advisable because, as seen at Fort Polk, the vendor may change or add equipment after the operation has started.

11.2.6 Plant Operation

A smooth field operation has its basis in a good bench-scale study and design. However, once the plant is on site, the following considerations are required to ensure smooth operation:

☐ Adequate Number and Qualification of Operators. The number of on-site operators should be sufficient to keep all elements of the plant operating in tandem. The operator requirement may depend to some extent on the degree of automation built into the plant. An on-site process chemist and an on-site AA unit are desirable to help maintain adequate process control.

❏ Pretest the Plant for Leaks. After the plant is assembled on site, water should be run throughout the entire plant to detect and fix leaks. This should be done before soil and process solutions are run through the plant to minimize potentially hazardous runoff.

11.2.7 Process Verification

During the operation, the site will have to verify that processing goals have been achieved. The following sampling and analysis issues should be considered to obtain meaningful results:

❏ Large composite samples of the processed soil (between 150 to 300 lb) should be collected periodically. The composite should be collected by taking several grab samples from the processed soil pile, or by collecting periodic grab samples from the processed soil discharge conveyor.

❏ Either the entire composite sample may be processed and analyzed, or multiple grab samples may be collected (by coring from the composite sample) and analyzed separately. Collecting multiple 1-L core samples does not require special sampling equipment and is likely to be more economical for most sites. Both methods provide reasonably good estimates of the total and TCLP metals concentrations.

❏ Each 1-L sample sent to the laboratory should be dried and run through a sample splitter to obtain multiple 100-gram aliquots for TCLP analysis. The rest of the material should be reduced in size to 100 or 200 mesh by grinding. Multiple 8-gram aliquots of the ground material can be collected by running it through a sample splitter. The 8-gram aliquots can be microwave-digested for AA or ICP analysis.

❏ Because of the special sample preparation and the standard 18-hr TCLP extraction requirements, laboratory verification will require at least 4 to 5 days after the sample is collected, assuming that the entire sample preparation and analysis chain proceeds smoothly and the site can bear the associated laboratory surcharges. This may mean that each batch of processed soil has to be stored at the site until verification is received for that batch. However, once the plant is at steady state, and initial verification has been done, future verification may be needed only occasionally, as long as the plant continues to operate at steady-state. If storage space on the pad is limited, the soil can be transferred to the berm while awaiting verification. If the location of the returned soil is marked it can be retrieved if, in a worst-case scenario, verification indicates TCLP or total metals criteria have not been met.

❏ Ideally, the site should make arrangements to store processed soil from at least 3 to 4 days of processing. If that is not possible, and if some small sample preparation equipment (oven, grinder, sample splitter, and sieve) can be mobilized at the site, at least a total metals analysis of multiple grabs from the processed soil could be done using an AA or XRF unit. This would provide some preliminary indication of process performance before returning the processed soil to the range.

11.2.8 Costs

Figure 11-5 summarizes the cost projections for application of this technology at small-arms ranges in comparison with other common options. For stabilization and physical separation/acid leaching, the projections are based on processing the soil to meet the TCLP lead criterion. Both stabilization and physical separation/acid leaching are on-site technologies and therefore will incur high fixed costs. At smaller sites, therefore, off-site landfilling is cheaper. However, with landfilling, the site undertakes transportation risk and potential long-term liability. At larger sites, stabilization and physical separation/acid leaching become less costly compared with landfilling.

Compared with stabilization technology, physical separation/acid leaching appears to be slightly more expensive regardless of site size. The reason for this is that stabilization technology involves simpler equipment and, therefore, lower fixed costs. Processing times for stabilization are usually shorter than for leaching, and this results in faster processing with equivalent-sized equipment. However, if a true cost-benefit analysis is done, physical separation/acid leaching may prove to be the most attractive option for small-arms ranges compared with either landfilling or stabilization, regardless of the site size, because of the following reasons:

☐ Physical separation/acid leaching removes heavy metals from the soil, increasing the range of beneficial uses available to the site. At active ranges the processed soil is suitable for reuse in the berm. At inactive ranges, the site may find other beneficial uses.

☐ Most of the recovered heavy metals are recycled and reused off site. This reduces potential long-term liability. This solution may be more attractive to regulators as well, making implementation of physical separation/acid leaching technology easier.

☐ Process plants and operators using physical separation/acid leaching technology are available from several commercial vendors. The site does not have to purchase and operate the plant. The process can be implemented fairly quickly and an active range may not have to be shut down for extended periods of time.

In summary, the physical separation and acid leaching technology is an attractive option for small arms range maintenance or remediation.

11.2.8 Costs

Figure 11-5 summarizes the cost projections for application of this technology at small-arms ranges in comparison with other common options. For stabilization and physical separation/acid leaching, the projections are based on processing the soil to meet the TCLP lead criterion. Both stabilization and physical separation/acid leaching are on-site technologies and therefore will incur high fixed costs. At smaller sites, therefore, off-site landfilling is cheaper. However, with landfilling, the site undertakes transportation risk and potential long-term liability. At larger sites, stabilization and physical separation/acid leaching become less costly compared with landfilling.
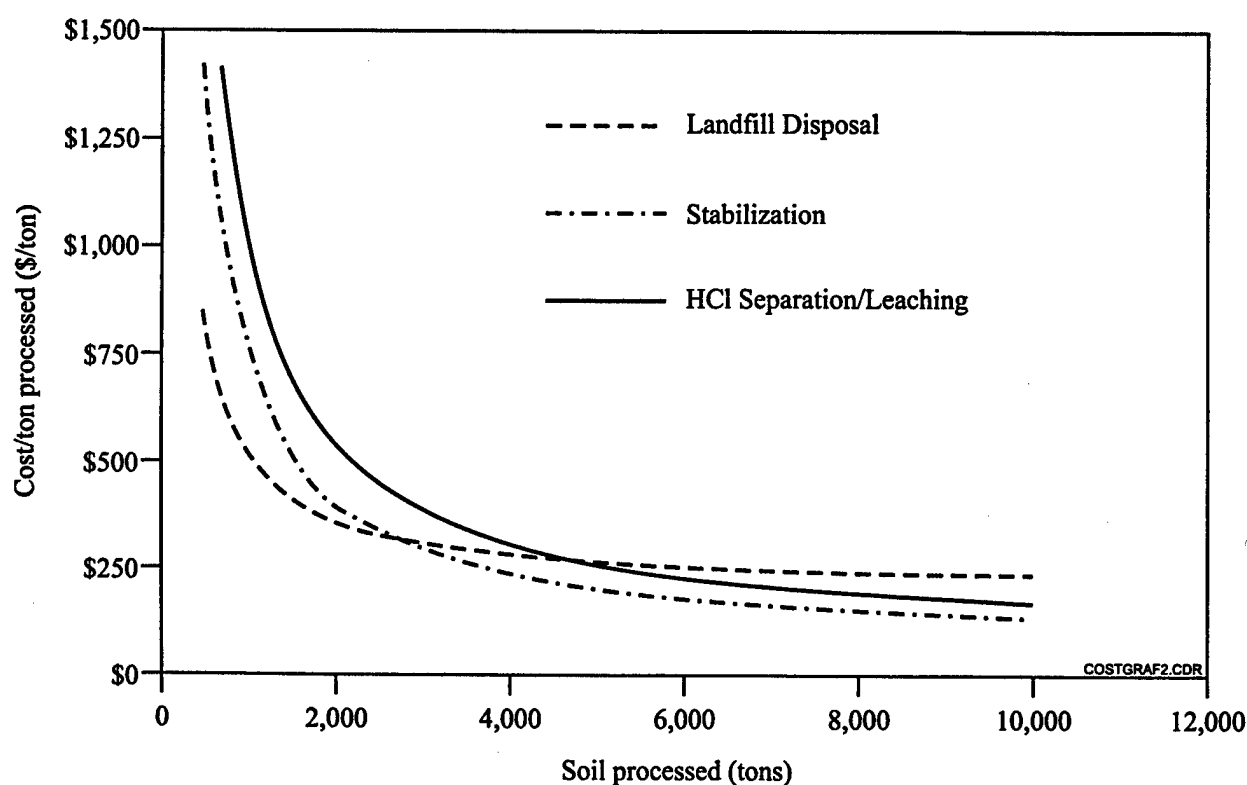

Figure 11-5. Cost Comparison of Alternative Technologies

Compared with stabilization technology, physical separation/acid leaching appears to be slightly more expensive regardless of site size. The reason for this is that stabilization technology involves simpler equipment and, therefore, lower fixed costs. Processing times for stabilization are usually shorter than for leaching, and this results in faster processing with equivalent-sized equipment. However, if a true cost-benefit analysis is done, physical separation/acid leaching may prove to be the most attractive option for small-arms ranges compared with either landfilling or stabilization, regardless of the site size, because of the following reasons:

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❏ Physical separation/acid leaching removes heavy metals from the soil, increasing the range of beneficial uses available to the site. At active ranges the processed soil is suitable for reuse in the berm. At inactive ranges, the site may find other beneficial uses.

❏ Most of the recovered heavy metals are recycled and reused off site. This reduces potential long-term liability. This solution may be more attractive to regulators as well, making implementation of physical separation/acid leaching technology easier.

❏ Process plants and operators using physical separation/acid leaching technology are available from several commercial vendors. The site does not have to purchase and operate the plant. The process can be implemented fairly quickly and an active range may not have to be shut down for extended periods of time.

In summary, the physical separation and acid leaching technology is an attractive option for small arms range maintenance or remediation.

12 References

Acar, Y.B., and A.N. Alshawabkeh. 1993. "Principles of Electrokinetic Remediation." *Environ. Sci.* Technol. 27(13):2638-2647.

Air Force Center for Environmental Excellence (AFCEE). 1992. *Remedial Technology Design, Performance, and Cost Study.* Environmental Services Office. July, 1992.

Arniella, E.F., and L.J. Blythe. 1990. "Solidifying Traps Hazardous Wastes." *Chemical Engineering, 97*(2): 92-102. February.

Baes, Jr., C.F., and R.E. Mesmer. 1986. *The Hydrolysis of Cations.* Robert E. Kreiger Publishing Co., Malabar, FL.

Battelle. 1996. *Technology Demonstration Plan for Joint Small-Arms Range Remediation at Fort Polk, Louisiana,* Battelle Columbus Operations, October 14.

BDM. 1997. *Demonstration of Physical Separation/Leaching Methods for the Remediation of Heavy Metals-Contaminated Soil at Small-Arms Ranges.* Prepared for Naval Facilities Engineering Service Center and U.S. Army Environmental Center, September.

Buelt, J.L., C.L. Timmerman, K.H. Oma, V.F. FitzPatrick, and J. G. Carter. 1987. *In Situ Vitrification of Transuranic Waste: An Updated Systems Evaluation and Applications Assessment.* PNL-4800. Pacific Northwest Laboratory, Richland, Washington.

Conner, J.R. 1990. *Chemical Fixation and Solidification of Hazardous Wastes.* Van Nostrand Reinhold, New York, New York.

DoD, see U.S. Department of Defense

DuGuay, T. 1993. "Reclaim Metals to Clean Up Soils." *Soils,* March 26, pp. 28-33.

Fix, M., and B. Fristad. 1993. "Lead is Washed from Soil at Twin Cities Army Ammunition Plant Site." *Superfund Week,* 7(44): 2-3.

Fristad, W.E., D.K. Elliott, and M.D. Royer. 1996. "EPA Site Emerging Technology Program: Cognis Terramet® Lead Extraction Process." *Air & Waste Manage Assoc.* 46:470-480.

Gy, P.M. 1982. *Sampling of Particulate Materials – Theory and Practice,* Elsevier Scientific Publishing Company, New York, NY.

Hazen, Inc. 1997. "Characterization of the Lead Content in a Sample of Fort Polk Soil," HRI Project 8939, March 21.

Heath, J.C., L. Karr, V. Novstrup, B. Nelson, S.K. Ong, P. Aggarwal, J. Means, S. Pomeroy, and S. Clark. 1991. *Environmental Effects of Small Arms Ranges.* NCEL Technical Note N-1836. Naval Facilities Engineering Command, Naval Civil Engineering Laboratory, Port Hueneme, CA. October.

Igew, G.J., P.D. Walling, Jr., and D. Johnson. 1994. "Physical and Chemical Characterization of Lead-Contaminated Soil." *1994 Innovative Solutions for Contaminated Site Management.* The Water Environment Federation conference, Miami, FL. March 6.

Johnson, J.L., D.D. Chirban, L.A. Karr, and J.C. Heath. 1994. "Heavy Metals Removal from Small-Arms Ranges." *Process Mineralogy XII - Applications to Environment, Precious Metals, Mineral Beneficiation,* Pyrometallurgy, *Coal and Refractories.* Eds. W. Petruk and A.R. Rule. The Minerals, Metals, and Materials Society, Warrendale, PA.

Kragten, J., Ellis Horwood Ltd. 1978. *Atlas of Metal-Ligand Equilibria in Aqueous Solution.* John Wiley & Sons, New York.

Krishnamurthy, S. 1992. "Extraction and Recovery of Lead Species from Soil." *Environmental Progress 11*(4):256-260.

Lanouette, K.H. 1977. "Heavy Metals Removal", *Chem. Eng., 84*(21): 73-80.

Lead Industries Association. 1992. *Lead* Recycling – *1992 Directory*. Lead Industries Association, New York, NY.

Lewis, M.L., N.P. Barkley, and J. McCoy. 1995. "1994 Update of U.S. EPA's Superfund Innovative Technology Evaluation (SITE) Emerging Technology Program." *Environmental Manager. 1*: 31-42.

Marks, R.E., Y.B. Acar, and R.J. Gale. 1992. "Electrokinetic Soil Processing: An Emerging Technology." *47th Purdue Industrial Waste Conference Proceedings,* Lewis Publishers, Inc., Chelsea, Michigan.

Martell, A.E., and R.M. Smith. 1974. *Critical Stability Constants*, Plenum Press, New York.

Means, R.S. 1996. Softbooks™ – Environmental Restoration Cost Books. ECHOS, LLC.

National Rifle Association (NRA). 1989. *The Range Manual: A Guide to Planning and Construction*. National Rifle Association, Fairfax, VA.

Paff, S.W., and B. Bosilovich. 1993. "Remediation of Lead-Contaminated Superfund Sites Using Secondary Lead Smelting, Soil Washing, and Other Technologies." In J.P. Hager, B.J. Hansen, J.F. Pusateri, W.P. Imrie, and V. Ramachandran (Eds.), *Extraction and Processing for the Treatment and Minimization of Wastes*, pp. 181-200. The Minerals, Metals, and Materials Society, Warrendale, PA.

Perry, R. H., and C. H. Chilton. 1984. *Chemical Engineers' Handbook*, 6th ed. McGraw-Hill Book Company, New York.

Pitard, F.F. 1992. *Pierre Gy's Sampling Theory and Sampling Practice, Volume I, Heterogeneity and Sampling,* CRC Press, Inc., Boca Raton, FL.

Prengaman, R.D., and H. McDonald. 1990. "RSR's Full-Scale Plant to Electrowin Lead from Battery Scrap." In T.S. Mackey and R.D. Prengaman (Eds.), *Lead-Zinc '90*, The Minerals, Metals & Materials Society (TMS). TMS Annual Meeting, Febuary 18-20, Anaheim, CA.

Probstein, R.F. and R.E. Hicks. 1993 "Removal of Contaminants from Soils by Electrical Fields." *Science, 260*:498-503.

Ramachandran, V., S.R. Gilbert, R.I. Cardenas, and M. Zwierzykowski. 1993. "Recycling Metal-Bearing Hazardous Waste." In J.P. Hager, B.J. Hansen, J.F. Pusateri, W.P. Imrie, and V. Ramachandran (Eds.), *Extraction and Processing for the Treatment and Minimization of Wastes,* pp. 131-141. The Minerals, Metals, and Materials Society, Warrendale, PA.

Superfund Week. 1993. "Lead is Washed from Soil at TCAAP Site." *Superfund Week 7*(44):5.

Taggart, A.F. 1945. *Handbook of Mineral Dressing, Ores and Industrial Minerals*. John Wiley & Sons, New York, NY.

Testa, S.M., and D.L. Patton. 1994. "Soil Remediation via Environmentally Processed Asphalt™ (EPA)." In J.P. Hager, B.J. Hansen, W.P. Imrie, J.F. Pusateri, and V. Ramachandran (Eds.), *Extraction and Processing for the Treatment and Minimization of Wastes 1994,* pp. 461-485. The Minerals, Metals and Materials Society, Warrendale, PA.

U.S. Department of Defense, Military Handbook. 1992. "Range Facilities and Miscellaneous Training Facilities Other Than Buildings," "MIL-HDBK-1027/3B, 30 November.

U.S. Environmental Protection Agency. 1990. *Handbook on In Situ Treatment of Hazardous Waste-Contaminated Soils*. EPA/540-2-90-002. U.S. EPA, Office of Research and Development, Risk Reduction Engineering Laboratory.

U.S. Environmental Protection Agency. 1991. *Selection of Control Technologies for Remediation of Lead Battery Recycling Sites*. EPA/540/S-91/014. Office of Research and Development, Washington, DC.

U.S. Environmental Protection Agency. 1991. *Superfund Engineering Issue – Treatment of Lead-Contaminated Soils*. EPA/540/2-91/009. Office of Solid Waste and Emergency and Remedial Response, Washington, DC; and Office of Research and Development, Risk Reduction Engineering Laboratory, Cincinnati, OH.

U.S. Environmental Protection Agency. 1992. *Engineering Bulletin: Selection of Control Technologies for Remediation of Lead Battery Recycling Sites*. 18 pp. EPA/540-S-02/011. Office of Emergency and Remedial Response, Washington, DC.

U.S. Environmental Protection Agency. 1992. *Potential Reuse of Petroleum-Contaminated Soil: A Directory of Permitted Recycling Facilities*. EPA/600/R-92-096. Report prepared for the U.S. EPA Office of Research and Development by J.H. Nash, S. Rosenthal, G. Wolf, and M. Avery of Chapman, Inc. and Enviresponse, Inc. 38 pp.

U.S. Environmental Protection Agency. 1992. *The Superfund Innovative Trechnology Evaluation Program – Technology Profiles*, 5th ed. EPA/540/R-92-077. Office of Research and Development, Risk Reduction Engineering Laboratory. Cincinnati, OH.

U.S. Environmental Protection Agency. 1992. *Vitrification Technologies for Treatment of Hazardous and Radioactive Waste*, EPA/625/R-92/002, U.S. EPA Office of Solid Waste and Emergency Response, Washington, DC.

U.S. Environmental Protection Agency. 1993. *Technical Resource Document: Solidification/ Stabilization and its Application to Waste Materials*. EPA/530/R-93/012. Risk Reduction Engineering Laboratory, Cincinnati, OH.

U.S. Environmental Protection Agency. 1994. *Superfund Innovative Technology Evaluation Program C Technology Profiles*, 7th ed. EPA/540/R-94/526. Office of Research and Development, Washington, DC.

U.S. Environmental Protection Agency. 1994. *VISITT C Vendor Information System for Innovative Treatment Technologies*. EPA/542/R-94/003 (with update to Version 4.0). Office of Solid Waste and Emergency Response, Washington, DC.

U.S. Environmental Protection Agency. 1995. *Contaminants and Remedial Options at Selected Metal-Contaminated Sites*. EPA/540/R-95/512. Office of Research and Development, Washington, DC.

Van Benschoten, J.E., M.R. Matsumoto, and W.H. Young. 1997. "Evaluation and Analysis of Soil Washing for Seven Lead-Contaminated Soils." *Journal of Environmental Engineering, 123*(3):217-224.

Valenti, M. 1992. "Recovering Heavy Metals." *Mechanical Engineering*, December, pp. 54-58.

Wethington, A.M., A.Y. Lee, and M.G. Gorman. 1992. "Decontamination of Lead Wastes from Superfund Sites." *Proceedings of the Hazardous Materials Control Resources Institute (HMC)-South '92 Exhibitor Conference & Exhibition*, New Orleans, February 26-28, pp. 117-122.

Wills, B.A. 1985. *Minerals Processing Technology*, 3rd ed. Pergamon Press, New York, NY.